

THE PILOT PLANT REAL BOOK

**A Unique Handbook for the
Chemical Process Industry**

Francis X. McConville

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**FXM ENGINEERING AND DESIGN
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Published by:
FXM Engineering and Design
6 Intervale Road
Worcester MA 01602
www.fxmtech.com

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ISBN Printed First Edition
First Printing 2002

Publisher's Cataloging-in-Publication
(Provided by Quality Books, Inc.)

McConville, Francis X.

The pilot plant real book : a unique handbook for the
chemical process industry / Francis X. McConville.—

1st. ed.

p. cm.

Includes bibliographical references and index.

LCCN 2002108236

ISBN 0-9721769-1-8

1. Chemical plants—Pilot plants—Handbooks, manuals,
etc. 2. Chemical processes—Handbooks, manuals, etc.

3. Chemistry, Technical—Handbooks, manuals, etc.

I. Title

TP155.5.M33 2002

660'.28072

QBI02-200782

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Author's Preface

Is it more efficient to operate a distillation at atmospheric pressure or under vacuum?

What's the difference between a single-phase and a three-phase motor?

What's a Haz-Op?

What's a Class 1, Div. 1 solvent?

What's the difference between a thermocouple and an RTD?

What's an intrinsically safe circuit?

What are the best solvents for azeotropic removal of water?

What happens to an azeotrope under vacuum?

What's a Friedel-Crafts reaction?

These are typical of the questions I often faced as a process engineer fresh from graduate school – questions easily answered by someone experienced in the field, but which frequently sent me digging into a veritable library of books and articles on wide-ranging subjects to get a simple answer. So much fundamental information that I thought should be at my fingertips lay deeply buried in huge handbooks or obscure references.

Thus was the idea for this book born. I have endeavored to collect here, in one convenient place, the information most often called for by chemists and engineers working in process development. Hence the volume is rich with physical property data, information on processing equipment, formulas, tips and techniques, and safety recommendations. Monographs discuss many important aspects of chemical processing and development, and pilot plant operation. Materials and piping data help ensure that “jury-rigging” temporary pilot setups need never compromise safety.

But this has evolved from a mere handbook to a work that I hope will also help fill a major gap between those who discover new chemical processes and those whose job it is to scale them up to commercialization. Because of what I see as a failure of the education system today, graduating chemists and engineers are often ill-prepared for the unique challenges that are part and parcel of process scale-up. Therefore, I have included detailed suggestions for developing chemical processes that will be more easily transferred from lab to plant, lists of do's and don'ts in chemical development, and advice for improving communication across the development organization.

When asked by a colleague who my intended audience was, I had to confess that I wrote the book for myself – and for technology transfer specialists just like me whose responsibilities straddle the fence between chemistry and engineering. Process development is a highly interdisciplinary effort and the most successful processes are developed through close cooperation between various areas of technology. Chemists, for example, need to have an appreciation for phenomena such as heat transfer and mixing effects to develop scalable processes, whereas some “chemistry” problems can be solved, and some routes possibly rescued, by creative engineering solutions. The development chemists are intimately aware of the critical processing steps, and the experienced engineers understand the limitations of plant equipment. Communication is key to keeping the team working towards a common goal, and the broader a person's perspective is, the more valuable will be his or her input. There is a definite advantage to being a “jack-of-all-trades” in the field of process development.

It's often an educational, and sometimes surprising, experience for a chemist or engineer to bring a carefully tuned process to the floor of the pilot plant for the first time – to “scale it up” as we say. Things do not always scale up as expected. Operations are usually much more involved and take much longer than at the lab bench. The potential hazards are greater, as are the economic consequences of a failed batch. And this works both ways. Technicians who operate pilot plant equipment may be baffled by the chemistry of the process they are operating. But a broad knowledge

base is the common denominator that can bridge the gap and facilitate what can sometimes be a very involved process.

Although this book is specifically geared to development and scale-up personnel, there is enough material here of general technical interest to make the book useful to anyone involved in the chemical sciences. In an effort to reach a broad readership, I have tried to keep the complex mathematics and engineering treatments to a minimum, and provided references for further study as appropriate.

The Table of Contents on page v will help familiarize readers with the overall content of the book. Since my guiding principle has been to make it easy for readers to find what they need, I have also included more detailed tables of contents for each individual chapter, an exhaustive index, and section tabs as guides. Bibliographic references and recommended reading list follow Chapter 11. References are indicated by bracketed numerals throughout the text. A list of common governing agency acronyms is also provided in Chapter 11.

I apologize in advance for the use of English Engineering units, which, unfortunately, are still widely used in many areas of American technology. I have attempted to provide both metric and English units where space and practicality allowed. The unique format of the conversion factors provided in Chapter 11 should simplify conversion between unit systems. I also apologize for any repetition. Some is deliberate, since I feel that certain important concepts bear repeating.

Readers are encouraged to report errata or omissions, fill in holes in the data, download templates, check for updates, or make suggestions for future editions at our website www.pprbook.com. Please contact me through the site if you feel you have information that would make a useful addition. Your comments are always welcome.

The name? For decades, aspiring young jazz musicians who wanted to sit in on sessions had to master the “Real Book”, a bootleg, photocopied collection of the great jazz standards – all the songs anyone needed to know in one place. I hope that, in the same spirit, this book will put at your fingertips the information you need to help you perform your work more efficiently.

Thanks for reading.

–Francis X. McConville

Acknowledgments

I would like to acknowledge a number of people who guided and advised me along the way: my friend and colleague, Walter Crockett, for his editorial comments and encouragement; my technical editor, Jon Thunberg, formerly of Hampshire Chemical Corporation; my proofreader, Janice Morgan Jones; my former colleagues, Robert Prytko, Kostas Saranteas and Roger Bakale of Sepracor, Inc., for their suggestions and additions; the many organizations who offered information or granted me permission to reprint material – MG Industries, Victor Specialty Products, Pfaudler, Inc., Dow Chemical Co., H. S. Martin Glass Co., Büchi GlasUster, Chemical Transfer Partnership Corp. and Martell Associates; the folks at Mercantile Press; all the family and friends who offered their support; and especially my wife, Evelyn, for her infinite patience.

Contents

1 The Pilot Plant

Role of the Plant • Factors in Scale-Up • Some Do's and Don'ts • Tips for Developing Scalable Reactions • Calorimetry/Safety Screening • Haz-Ops • Flow Diagrams • Batch Records • GMP

2 Equipment and Operations

Equipment Train • Reactors • Agitation • Raw Material Charging • Reaction Control • Sampling • Workup • Distillation • Crystallization • Isolation/Filtration • Drying • Equipment Cleaning

3 Liquid Handling

Pumps • Pump Sizing, Selection and Troubleshooting • Pipes, Fittings and Valves • Flanges and Gaskets • Hose • Friction and Viscosity Effects • O-rings • Stoppers • Liquid Storage Vessels

4 Heat Transfer

Heat Transfer in Stirred Vessels • Heat Exchangers • Temperature Control Units • Heat Transfer Fluid Selection and Properties • Glycols and Brines • Steam • Process Chillers

5 Electricity and Instrumentation

Electrical Safety • Electric Power Basics • Receptacle Types • Electrical Enclosure Data • Intrinsic Safety • Motors • Temperature, Pressure and pH Measurement • Process Control Basics

6 Solvents

Safe Handling • Properties and Selection • Solubility Map • Temperature/Vapor Pressure Relationships • Distillation • Binary Azeotropes • Ternary Water-Containing Azeotropes • Water Data

7 Compressed Gases

Safe Handling • Physical Properties • Cryogenic Liquids • Cylinder Specifications • Connection Data • Pressure Regulators • Gas Metering • Air Data • Air Compressors • Vacuum Systems

8 Chemical Data

Properties of Acids and Bases • Dilutions • pKas • pH Buffers • Color Indicators • Aqueous Solubility Data • Aqueous Solution Density • Organic Functional Groups • Reaction Types

9 Chemical Hygiene and Safety

Safe Handling of Chemicals • MSDSs • Hazards Classifications • Labeling • Fire Safety • Personal Protective Equipment • Glove Selection • Respirators • Incompatible Chemicals

10 Materials Selection

Properties of Elastomers, Plastics, Metals and Glass • Metallic Corrosion Data • Glass Corrosion • Material Compatibility Table

11 Miscellaneous

Unit Conversion Tables • Physical Constants • Mathematical Relationships • Geometric Formulas • Temperature/Pressure Conversion Charts • Cold Mixtures • Sieve Sizes • Agency Acronyms

Bibliography

Additional Recommended Reading

Index

SEE DETAILED TABLES OF CONTENTS AT EACH CHAPTER

1 The Pilot Plant

Contents

The Role of the Pilot Plant	1-2
Major Factors in Scale-Up	1-3
12 Things to Do During Process Scale-Up	1-5
12 Things to Avoid During Process Scale-Up	1-6
Developing Scalable Reactions	1-7
The Role of Calorimetry and Process Safety Screening	1-9
The Haz-Op	1-10
The Batch Checklist	1-12
The Process Flow Diagram	1-13
The Batch Record	1-14
The Campaign Report	1-15
Notes on GMP	1-16

The Role of the Pilot Plant

A chemical pilot plant can be many things to many people. It can range in scale from a room with a walk-in fume hood and some 20-liter glassware to an experimental petroleum refinery. This book focuses on the relatively small-scale development operations found in the pharmaceutical and fine chemical industries. These usually entail manual batch operation of processes that are often not fully defined, in standard chemical reactors from, say, 20 liters to 200 gallons. Even with the increasing role of automated bench-scale reactors in process development, most processes will be tested in the pilot plant before reaching commercial scale. The jump from bench to pilot plant will usually be the single largest numerical increase in scale (perhaps up to 100 times) which the fledgling process will ever experience, and therefore can present the greatest challenges.

But “scale-up” is only one of the ways in which a pilot plant can prove itself valuable. It can play a major role in streamlining what can often be a lengthy development process. Here are some important functions that a pilot plant can perform in process development:

- Produce raw materials and intermediates to supply further development work.
- Produce developmental quantities of new compounds for evaluation, toxicity testing, safety and stability studies, clinical trials, and introduction into the market. Often the first few kilograms of a new compound are the most difficult to make, since an optimized route has not yet been developed.
- Demonstrate that processes can be successfully scaled up and that there are no unexpected ramifications of extended operating times, slower rates of addition or mixing effects at larger scale.
- Ensure that no important details have been overlooked.
- Test the effects of using commercial-grade raw materials and solvents.
- Identify the best ways to handle and analyze reactants, intermediates, products, waste streams and off-gases.
- Check the effect of the buildup of impurities in recycle streams and other long-term effects.
- Test materials of construction.
- Complete a more detailed mass balance, and obtain better estimates of yield and effluent stream generation.
- Help to better estimate process costs and increase management confidence in investing for full-scale production.
- Obtain design data and optimum operating parameters for specifying larger-scale equipment.
- Train members of the technology transfer team preparing for commercial production.
- Help develop a comprehensive and detailed operating procedure for transfer to manufacturing.

Often, using large-scale glassware is the first scale-up step, but this can be extremely dangerous if mixing is not reliable and if the glassware must be handled manually. It is not easy to cool such equipment, and a spill can be a disaster resulting in exposure to flammable solvents or toxic gases. These issues are much more easily controlled using properly designed glass or stainless reactors in an explosion-proof environment.

Scaling up a chemical process early in its lifetime can be helpful in identifying potential scale issues that may require engineering assistance or special equipment to handle, in identifying the rate limiting steps, and in giving a feel for the overall workability of the process. Scaling up too early, of course, risks process changes and wasted time and resources. Involving team members of various disciplines early reduces the likelihood that process changes will be needed later.

For countless reasons, investment in proper pilot facilities is well worth it. Often, the pilot plant will soon prove itself so useful that it won't be able to keep up with the demand. Flexibility is one of the keys to success. Priorities shift, projects are added or dropped and synthetic routes are often altered on the fly. The pilot plant must be able to adapt. The facility should be able to handle a variety of processes safely with a minimum of special equipment. Equipment should offer wide corrosion resistance, and should be easy to clean to prevent cross-contamination between batches and products. Space should be available for experimental setups. And pharmaceutical pilot plants need to accomplish all this within the added constraints of GMP operation (see page 1-16). The following pages explore some important thoughts and concepts that will help you maximize the utility of your pilot plant and take full advantage of its tremendous value.

Major Factors in Scale-Up

Scaling up chemistry from laboratory glassware to larger reactor vessels is by no means a simple linear process. A number of things that may not be immediately evident to the inexperienced scientist are very different at larger scale, and many of these can have a tremendous impact on process performance. The biggest problem areas faced by the scale-up team are listed below, along with a brief explanation of how they can affect the success of the project. It might seem that by maintaining dynamic similarity during scale-up (keeping the same mixing velocities, temperatures, concentrations, etc.) performance would be equivalent, but this is far from the case. The advantages of getting process engineers involved early in the development effort may become obvious reading through the following items. More detailed discussions of techniques to circumvent these difficulties can be found on page 1-7 and throughout Chapter 2.

Expanded Time Scale – Perhaps the biggest surprise to the chemist or engineer bringing a process to the plant for the first time is how long everything takes. Charging the raw materials and solvents can take hours. A heating or cooling step accomplished in minutes at the bench can take 8 hours or longer. A simple distillation can take 12 hours. Isolation of a product in a centrifuge can take more than 24 hours at large scale. A laboratory synthetic step that a chemist could complete in a workday and have the product in the drying oven by dinnertime can take 2-3 days of round-the-clock operation to complete. Such extended processing can cause decomposition, polymorphic shifts or other problems. Stream stability is an important aspect of scale-up that should be borne in mind throughout the development process.

Chemical Hygiene – Handling chemicals at the large scale poses greatly increased risks and chances for exposure to toxic substances. Laboratory fume hoods can cover many sins, but at the pilot scale, electrostatic hazards of handling dry powders, flammable solvents and wet filter cakes are greatly increased. Dusting is a huge problem when transferring bulk solids. Off-gases must be treated using a scrubber to prevent escape into the atmosphere. All of this means that various personal protective equipment must be used, and safety devices and interlock systems must be installed.

Heat Transfer – Laboratory flasks have a relatively high surface-to-volume ratio. This, and their small size, makes it easy to heat or cool them quickly (think ice baths) and to easily hold the reaction temperature constant. Not so in larger reactors, where heat transfer surface area/volume is greatly diminished, and where heating and cooling must be accomplished by means of a heat transfer medium pumped through a jacket or heating coil. Heat removal rates can be 10 times slower per unit volume at the pilot scale, and as much as 30 times slower at commercial scale. This approaches adiabatic operation, which should hint at the value of reaction calorimetry in process safety screening, especially for exothermic reactions (see page 1-9). The heat generated per unit volume in an exothermic reaction remains constant as scale increases. However, the total amount of heat to be removed increases as the cube of the reactor size, whereas, for jacketed reactors, the area available for heat transfer increases only as the square of the reactor size (see page 4-4). Other factors can also affect heat transfer, namely characteristics of the heat transfer fluid, the thickness and thermal conductivity of the reactor walls, and mixing effects. Low temperature reactions also necessarily take much longer because of these limitations.

In order to accomplish heating in a reasonable time frame, process operators must try to maximize the temperature difference between the vessel jacket and the vessel contents. Wall temperatures might exceed temperatures experienced by the reaction mixture at the bench, raising concerns about stability, e.g., decomposition on dry reactor walls. For cooling, jacket temperatures may be far lower than at the bench, which can lead to reagent freezing, crash crystallization and other effects not seen at smaller scale.

Temperature Control – Maintaining constant temperature is also much more difficult at larger scale. Constant temperature baths are not available. The temperature control system for a chemical reactor consists of a dynamic feedback loop to control the circulation of heat transfer fluid or some other medium. Heat generated in the reactor must be removed at a rate matching its production. Even an operation as simple as solvent addition while maintaining temperature can become a lengthy operation if, for example, the solvent is much colder than the batch or if there is a significant heat of dilution. Also, if the role of mixing is not well understood, blend-times may be longer, and temperature gradients may exist from the center to the edge of reactor, possibly increasing the rate of side reactions and the formation of by-products.

Reactor Mixing – Simply because of the increased geometry at large scale, blend times or turnover times can be significantly longer than at the bench. Blend times on the order of 1-2 seconds are typical in a laboratory flask, but may be

many times longer at larger scale. Local areas of excess reagents may exist, which can cause formation of by-products. In this way, reaction selectivity or yield can be affected. pH control is a perfect example of this phenomenon. Local high concentrations of acid or base at the point of addition can induce hydrolysis or other degradation reactions.

The impact of mixing effects is even more pronounced for two-phase reactions where interfacial surface area limits mass-transfer and can thus determine the reaction rate. Phase transfer reactions and extractions are good examples. Generating the same interfacial area per unit volume at larger scales is not trivial. Heterogeneous catalytic reactions face the same difficulties. Good suspension and distribution of the catalyst and high fluid velocities can have a significant impact on rate. If the rate drops too far, dangerous accumulations of unreacted reagents can occur. Viscosity, which is a strong function of temperature, also plays a role. This is why, in general, it is easier to scale up homogeneous reactions than heterogeneous ones. However, even some homogeneous reactions can be agitation-limited if reactants are not well mixed at the molecular level.

Mixing cannot be scaled up by simply using geometrically similar, but larger, mixers. Many other factors must be considered. For example, consider the case of a reaction catalyzed by a shear-sensitive enzyme. Operating a mixing impeller which is say, 5 times greater in diameter, at the same rpm as its bench-scale counterpart will result in an agitator tip speed, and thus a shear rate, which is also 5 times greater. But operating at 1/5 the rpm to obtain the same tip speed will not generate the same degree of turbulence. For a more detailed discussion of mixing scale-up, see Chapter 2.

Operating Volume – Evaporating to dryness, or very low volumes, is a convenient laboratory technique. This is usually not possible at scale because most reactors have maximum mixing levels of about 10-20% of their full capacity.

Reaction Control – Very often, because of the heat-removal limitations discussed above, processes are designed so that reaction rate can be regulated by controlled addition of a limiting reagent. But here, again, a greatly expanded time-scale must be expected at the pilot scale as compared to the bench. As mentioned above, for very low-temperature reactions the sensible heat of the reagent solution alone can approach the heat of reaction. Addition may require several hours, which can affect reaction equilibrium, yield and impurity formation.

Drying – Drying is often accomplished using vacuum ovens in the lab. Similar units (vacuum tray dryers) are also used in pilot and commercial plants, but the drying cycles will be much longer. Dynamic units such as rotary cone dryers, tumble dryers, paddle dryers and the like are also common but these types of equipment produce materials with very different physical characteristics than those obtained from tray driers. Differences in particle size distribution, bulk density, flowability, compressibility, etc., can all have a dramatic impact on the character of the product.

Raw Material Charges – Because of handling difficulties, it may not always be possible to be as accurate in charging raw materials at large scale as at the bench. Scale-up will require that acceptable charge ranges be determined, and the limits of these ranges must eventually be tested. Remember that pilot plant water and solvents may also be much colder.

Visibility – Chemists who rely on visual indication of reaction milestones, such as initiation of crystallization, may be disappointed to find that the best they can do at scale is to peer into a pitch-black reactor through a small sight window. Batch progress needs to be marked by quantifiable properties that can be measured using dependable analytical methods.

Reactor Access – Opening a reactor to add seed crystals or to collect samples is often not possible at large scale, because it may compromise batch integrity or operator safety. Often, it is necessary to use closed sampling systems (see page 2-15), solids addition chambers, or to add seeds in a slurry.

Work-Up – Often, pilot extractions and phase separations leave much more water behind than on the bench because of the greater tank surface area and longer settling times. Water removal can become more of a headache, and azeotropic drying or the addition of drying agents may become necessary. Waste minimization becomes an important concern during this sometimes volume-intensive stage of a process.

In general, liquid-phase batch processes are the easiest to scale up. It is simpler to control batch processes than continuous reactors, and the residence time is better-defined. Of course, continuous processing is sometimes necessary, especially for unstable products or highly exothermic reactions. The disadvantages of operating in the batch mode are the long downtimes between batches, the high labor requirements, and possible batch-to-batch variation in product quality. To minimize downtime and obtain consistent results, it is important to plan carefully and to understand, as well as possible, those process parameters that affect yield and quality.

12 Things to Do During Process Scale-Up

Below is a list of recommendations that can help you achieve safe and efficient operation in the kilo-lab or pilot-plant and reap the maximum benefit from the scale-up experience. Many of these things are expected practice in cGMP facilities, but regulatory requirements aside, adopting these suggestions will ultimately save time, improve the effectiveness of the plant as a development tool, and may significantly streamline the process development cycle.

- 1** – Develop an overall operating philosophy and guidelines for the minimum documentation required before new processes can be run in plant equipment – for example, a detailed Laboratory Process Description, Process Safety Information package, etc. Get management buy-in and support so that you can enforce these requirements.
- 2** – Set up operating and maintenance log books for each major piece of equipment in the plant (reactors, filters, dryers, pumps, hoses, etc.). In them, document all batches, cleaning operations, any tests made and their results, and any maintenance performed, beginning with the date of installation. Lab notebooks work well for this.
- 3** – Set up a sample log book. Using any reasonable numbering system, list in it every single sample collected in the plant for testing or retain. Include batch number, step number, time collected, purpose, test results, etc. This will become an invaluable archive for future reference, and will help ensure that important data is not lost. There may never be another opportunity to generate many of the samples collected during scale-up batches.
- 4** – Keep retain samples of all isolated products or intermediates produced in the kilo-lab or pilot plant. Store them in a cool, dark, dry place, or as appropriate for the particular material.
- 5** – Get engineers involved early in process development and route selection. The best processes result from a collaboration between those who best understand the chemistry and those who best understand pilot equipment limitations.
- 6** – Try to fix the process well prior to scale-up, so that there is time to focus on ensuring that the batch can be scaled up safely.
- 7** – Perform a hazards and operability review (Haz-Op) each time you bring a new process into the plant. This should include a detailed review of process safety information, contingency plans and emergency preparedness. See page 1-10 for more information on Haz-Op reviews.
- 8** – Insist on calorimetric testing to determine the stability of components, heats of reaction, the potential for decomposition and magnitude of thermal runaways. Most adverse events in the chemical process industry occur because of poorly understood chemical reactivity or insufficient heat removal.
- 9** – Create a written batch record (or “batch ticket” or “batch log sheet”) for each batch you conduct. Master records should be reviewed and signed by representatives of the departments involved (R&D, Engineering, etc.). Have a change-control system to track revisions and ensure that the most recent version is in use. See page 1-14.
- 10** – Use technical grade raw materials, or materials obtained directly from proposed large-scale suppliers – they will be more representative of future manufacturing sources.
- 11** – Always perform a bench-scale use test using the actual lots of all raw materials and in-house intermediates earmarked for the pilot batch. Ensure that the product made meets specification. It’s worth the time. If a pilot batch gives unexpected results, you’ll be able to eliminate raw materials as a source of the problem.
- 12** – Make the maximum use of each batch. Take as many in-process samples as possible and retain a good number for later troubleshooting. This includes key effluent streams. Use the opportunity to collect mass balance data, to test your energy balance relationships, and to verify analytical methods. Document all significant details in a campaign or batch report to make the information readily accessible at a later date. See page 1-15 for more on documenting a campaign.

12 Things to Avoid During Process Scale-Up

Getting pilot-plant personnel involved in process development early on can help avoid some common pitfalls that often hinder the technical or economic success of a batch campaign, and that may negatively affect safety. Below is a list of common methods or approaches which should be avoided because they are difficult to scale up, or because they are simply unsafe at anything larger than laboratory scale. It is not always possible to avoid all of these techniques, but raising the awareness level can fuel the search for more scalable alternatives. This is only a brief list – more detail is provided in following sections.

- 1** – Avoid reactions that require highly specialized equipment, or that are known to be hazardous and require special safety facilities, such as nitration reactions. At a minimum, obtain full calorimetric data and compare it to the maximum heat removal rate of the reactor. If necessary, consider tolling out such reactions to companies that specialize in them.
- 2** – Avoid "all-in and heat" operation – adding all reagents to the vessel and then heating up. While this is usually fine at the lab bench, possible exothermic reactions can create a thermal runaway situation at larger scales where heat removal rates are much slower. At unexpectedly high temperatures, exothermic decomposition reactions can take over, putting the reaction beyond any chance of operator control. Rapid gas evolution can compound the problem. It is better to design reactions so they can be controlled by slow addition of a limiting reagent at a rate that matches equipment cooling capacity. As a corollary to this rule, never add the catalyst to a reaction mixture last.
- 3** – Never heat a reactor without agitation. It can create hot spots that can erupt in violent boiling when the mixer is turned on. Never stop the agitator until a reaction mixture has cooled to a safe temperature.
- 4** – Do not operate a reactor less than 50°C from the known onset temperature of an exotherm that might run out of control.
- 5** – Avoid having to add solids to a reacting mixture. Manual addition can be extremely dangerous, and screws and conveyors for large scale solids addition are unreliable and expensive. Do not add solids to a hot or refluxing mixture.
- 6** – Never develop a process that relies on evaporating to dryness or to very low volumes. Although this is a useful laboratory technique, most large-scale reactors have a minimum mixing level about 20% of their capacity.
- 7** – Avoid reactions that must be isolated "immediately" such as the kinetic resolution of some enantiomers. At the manufacturing scale, product isolation alone can take up to 24 hours. Make sure the product slurry is stable for a sufficient length of time.
- 8** – Avoid using methylene chloride or other halogenated solvents that are considered environmental hazards. At the manufacturing scale, waste treatment or removal costs can often kill a process. Also see page 6-44 for a list of solvents limited for pharmaceutical use by the FDA. The use of phosphates should be minimized for similar reasons.
- 9** – Avoid hot filtrations or polish filtration of highly saturated solutions. Solids can crash out quickly in pipes or lines and clog the filter or other equipment. To prevent this in the plant, lines often have to be steam-traced, or preheated.
- 10** – Avoid reverse phase-splits (where the phase you want to retain is on the bottom). They require the addition of another vessel to the equipment train, a considerable expense at the manufacturing scale, and add time to the process cycle. Likewise, do not pour reaction mixtures into water to quench them. When scaled up, it will likely have to be the other way around.
- 11** – Try to avoid using chromatography for purification. While it is extremely useful in the lab, it is used commercially only for very high-value products or where there is no other choice. Even then chromatography requires very large amounts of solvents and support material. Better to develop a salt or other crystallizable form.
- 12** – Don't risk all of your raw materials or intermediates on one batch! Be prepared for the possibility that the batch, especially in a new process, may still hold surprises in spite of your careful preparations. Operator errors may also be more likely the first time through a new process.

Developing Scalable Reactions

Although cost is the bottom line for any commercial process, there can be a lot of flexibility in the way that the operation is approached. There is always a trade-off between cycle time, waste generation, the use of more or less hazardous reagents, and other choices. During the early stages of process development, it's difficult to tell which will be the most economical process in keeping with the principles of environmental responsibility and personnel safety. And without some experience at the pilot scale, an economic model is sometimes of limited use.

The first step, then, should be *getting to the pilot scale*. To develop processes that can be physically carried out in pilot equipment, safely and with equivalent yields and product characteristics, researchers must be attentive to the limitations faced when operating at that scale. It will help to review the things to avoid in process development (page 1-6), and the major factors that make pilot scale different from lab scale (page 1-3).

Get the input of the scale-up team early in process development. The value of doing so is often overlooked. A simple engineering solution, which might not be traditionally attempted at the bench, can rescue some promising routes from being abandoned. Conversely, experienced engineers might recognize at once that a proposed route must be modified to operate safely in plant equipment. Some examples:

- A reaction might actually be more easily controlled if it is run at a *higher* temperature, say at reflux, where there will be no accumulation of unreacted intermediate, and the rate of heat removal can be much greater than available from jacket cooling alone at lower temperatures.
- A quick pressure-filter experiment at the bench may demonstrate that a particular crystallization slurry will not filter at scale – crystallization studies, or alternative crystallization methods such as reverse addition, temperature cycling or slurry aging may be possible fixes.
- A high-yielding but exothermic route could be salvaged by using calorimetry to determine the heat of reaction and to calculate the addition rate of limiting reagent that will match plant heat-removal capacity.

Listed below are some thoughts and considerations, many of which will be familiar to the veteran development chemist, but which are nonetheless worth listing here as a reminder and to inspire scale-oriented thinking. More detailed recommendations can be found in the various sections of Chapter 2. Also, the text on practical process R&D by Anderson, with many tips on route selection and reaction optimization, is highly recommended [11].

Process Efficiency – Try to “telescope” reaction steps, that is, to use an intermediate product stream directly in the next step without isolating the intermediate. This approach minimizes time-consuming isolations, reduces handling losses, and eliminates drying time. Chemically convergent routes are also more efficient since intermediates can be prepared in parallel. Do not strive for extremely high-purity intermediates. Such purity may not be necessary at the next stage.

Time Scale – Consider the effects of extended processing times. Identify safe hold points. Examine stream stability by holding various in-process samples at expected conditions for a day or more.

Toxicity – Consider the hazards of raw materials, intermediates and by-products, and any special handling methods that might be required at the plant scale. Consider the environmental, handling and storage hazards associated with effluent stream disposal. Try to find less hazardous alternatives whenever possible.

Reagent Addition – Consider the effect of addition rate, reagent concentration, or of different addition methods. The order of addition is also critical. All of these can affect reaction equilibrium, side reactions and the formation of by-products and impurities. Be aware that the rate of controlled reagent addition will very likely be lower on scale up.

Raw Material Charges – It may not always be possible to achieve the same precision at scale as at the bench. Acceptable ranges for raw material and reagent charges will thus have to be established, and ultimately tested before commercialization. It is a good idea to think about this early. A range of $\pm 2\%$ is typical for key raw materials and $\pm 5\%$ for non-key materials (solvents, etc.). Get in the habit of charging everything, including solvents, by weight, not by volume.

Inert Atmosphere – Blanket your reactions with nitrogen or other inert gas to eliminate the possible effects of oxygen. Pilot equipment will be purged of all oxygen when the reaction is scaled up.

Operating Limits – Think about maximum allowable ranges for variables such as batch temperature and pressure. Note

that reactor-wall temperature may be much higher or lower than the batch depending on the operation.

Operating Volume – Determine the maximum and minimum volumes experienced during the process. The process must eventually be operated in a reactor which has definite maximum and minimum mixing volumes. Most commercial reactors can mix down to about 20% of their maximum volume and still provide reasonable heat transfer.

Mixing Effects – Consider the effects of mixing during the reaction, workup and crystallization. During reagent addition or pH adjustment, local areas of high concentration could cause side reactions. Run experiments at two extreme mixing rates (very fast and very slow) to study the effect on reaction rate and therefore on the rates of heat and gas evolution.

Heat Transfer – Avoid quick-cooling reaction mixtures in ice-baths, especially as you approach the final operating process. It is not uncommon for cooling operations at the pilot or commercial scale to take hours, as opposed to minutes at the bench, and it is important to understand the stability of the stream. Perform at least one experiment with slow cooling. Try to keep operating temperatures between -40°C and 150°C, typically available in most facilities.

Reaction Hazards – Perform calorimetry and safety screening on your reaction mixture, raw materials, intermediates and products, especially for exothermic reactions or if the possibility of thermal decomposition exists. Heat transfer is limited at larger scales and may not be sufficient to remove the heat of reaction. Some ways to overcome this may be to control the reaction by slow addition of limiting reagent, run it at a higher temperature to increase ΔT for heat removal or run the reaction at reflux, which often removes heat better than a reactor jacket, or reduce concentrations to slow the reaction rate. Of these methods, reagent addition is the most widely used, but watch for the accumulation of unreacted reagent if the temperature is too low. A higher temperature may actually be safer.

Reaction Monitoring – Think about a sampling protocol or other method for monitoring the progress of the reaction and workup. Think about sample quenching and storage for later analysis. Do reliable analytical methods exist?

Effect of Water – Examine the effect of small amounts of water, which can be introduced in solvents, etc. Set a water spec for azeotropic or other solvent drying operations.

Stripping – For determining the endpoint of solvent-switch operations, provide a quantitative method such as GC or refractive index. The latter is a simple, effective method that is often overlooked.

Stability – Are reagent solutions stable? Do they need special storage conditions? Is the reaction mixture stable until quenched? Is the product stable once crystallized? Will attrition (breakage) occur if the slurry is stirred for a long time?

Work-Up – Work-up should try to avoid large volumes of water, switching phases, and secondary solvents. The reaction should be designed from the beginning to anticipate work-up. Avoid water reactive reagents if possible. Investigate the likelihood of emulsification, which can take hours to separate in large-scale equipment (see page 2-17).

Chromatography – Avoid using chromatography for a final process. Large scale columns are available for pilot use, but chromatography is usually not commercially viable except for high-value biological or chiral products.

Product Crystallization – Establish solubility data for the final product. Perform crystallization and filtration studies to determine if there will be isolation difficulties. Try to use a single solvent. Try to avoid reslurry, which can be labor-intensive. However, a good procedure for rework or recrystallization may be invaluable if a batch fails to meet spec. Consider solvent recovery or a second-crop crystallization. Screen the product for polymorphs, hydrates, and solvates.

Drying – Perform a drying study in a vacuum oven. It is simple and will help determine the major resistances to drying.

Equipment Cleaning – Plan ahead to make reactor cleaning recommendations – determine product solubility in likely cleaning solvents and ensure that analytical methods exist. Know the limits of detection for these methods.

Product Specifications – Determine requirements for product purity and other quality attributes. How much analysis is needed? Do analytical methods exist? Begin compiling quality data early to establish a baseline.

Critical Process Parameters and Quality Attributes – Try to foster thinking that considers and quantifies the effects of process changes on product quality and yield. Develop quantitative in-process controls early on. This is a requirement for commercialization of pharmaceuticals, but makes good scientific sense in any situation.

The Role of Calorimetry and Process Safety Screening

For many reasons, pilot plant chemistry is inherently more dangerous than bench chemistry. It is obvious that the consequences of a runaway reaction or ignition event will be more catastrophic the larger the scale. But it may not be so obvious that, without the proper safeguards, adverse events are inherently *more likely* to happen at larger scale. As outlined earlier, reduced heat removal capacity in large reactors is one of the primary reasons, but just as important are the hazards associated with handling bulk flammable solvents and bulk powders. Part of process safety screening includes gathering data on the hazardous properties of process materials and the reaction mixture itself. Below a few of the most common analyses are mentioned, all of which can be performed in-house with the proper equipment, or, if need be, performed by independent testing labs such as HEL, Inc.

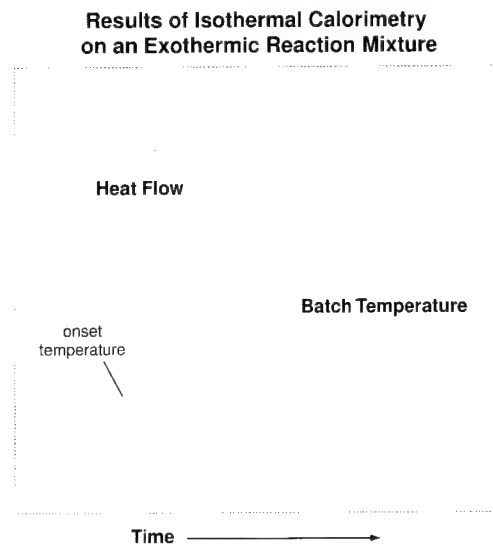
Differential Scanning Calorimetry (DSC) – This is a sensitive thermometric technique that can sometimes be used to approximate reaction onset temperature, to quantify the total energy released in an exothermic reaction, and to determine reaction mass specific heat. However, it is not easily used for reaction mixtures that must be agitated.

Reaction Calorimetry – Calorimetry, or thermal screening, is perhaps the most valuable and versatile tool for determining onset temperatures, heats of reaction, energy released, and specific heats, as well as the maximum temperature and pressure rise if there is a runaway reaction. Measuring exotherms is simple in principle, but to be meaningful, the determinations must be made carefully and with the proper equipment. Most calorimeters are equipped with stirrers and sensitive temperature control systems that enable measurement and control in a number of operating modes.

In **isothermal calorimetry**, the temperature of the test chamber is held constant or is carefully controlled to follow a preset profile. If the reaction mixture begins to generate heat, that heat is removed by a cooling system that measures how much heat is removed, from which the molar heat of reaction can be determined. The graph below shows the tracing from a hypothetical isothermal calorimeter experiment. In **adiabatic calorimetry**, the reactor is insulated so that no heat can escape to the environment. This closely mimics the poor cooling surface area in a large-scale reaction vessel. In this way, the reaction mixture is allowed to run uncontrolled to reach its maximum temperature, to the point of decomposition if it occurs. This is a worst-case scenario, and such tests often result in destruction of the test chamber.

In most calorimetric experiments, all the reactants are added to the chamber and it is slowly heated, but in other operating modes, the effect of reactant feed rate and other factors can be measured. For exothermic reactions, it is particularly valuable to know the heat of reaction per mole or kilo of limiting reagent. This fixes the maximum addition rate and the necessary jacket temperature to prevent thermal runaway and reagent accumulation in the reactor (see page 2-13).

Calorimetry has a number of other important uses. It is the preferred way to determine thermal stability of feeds and raw materials, intermediates and products. This can be augmented by **open cup testing** to determine if flammable gases are generated by reagents or a reaction mixture upon thermal decomposition. Calorimetry can also determine the total pressure generated (useful for sizing emergency pressure relief vents) and can quantify the thermal changes that accompany crystallization, which can help improve control options for that unit operation.



Powders and Dust Testing – Powder and dust explosions can easily be ignited by spontaneous discharge of static electricity that builds up in falling streams of solids and in drying filter cakes. Minimum Ignition Energy (MIE) and Minimum Ignition Temperature (MIT) are two tests that can help determine the likelihood of dust cloud ignition under the expected plant conditions. These data, along with the tendency of the material to become statically charged, the lower explosion limit (LEL) and the limiting oxygen concentration (LOC) for ignition are important in identifying any special handling precautions that may be needed. Shock-sensitivity testing is particularly important for grinding or blending operations. This type of testing basically consists of striking a sample with a special hammer-like apparatus that allows for observation of any ignition or detonation.

The Haz-Op

Many unforeseen things can occur when a chemical process is scaled up. Some can present serious safety issues, or at least, prevent smooth operation and threaten the success of the batch or campaign. For this reason, it is important to conduct a thorough review of the process, its potential hazards, and all proposed pilot-plant operations, before any chemistry is carried out. The pilot plant is not the place to experiment.

The obvious wisdom of performing a process review is backed up by certain legal requirements. OSHA regulations dictate that Process Safety Information (PSI) be collected for processes involving hazardous chemicals. This should include hazards data on materials used, MSDSs, reaction calorimetry results, identification of safe operating limits, the effect of deviations on the process, equipment information, etc.

The preferred way to conduct such a review is via the Hazards and Operability Study (Haz-Op), which usually consists of a meeting or series of meetings involving chemists, process engineers, plant supervisors, facilities personnel and others as needed. The Haz-Op provides a formal mechanism for dialogue and feedback about critical processing steps, potential pitfalls and specific equipment limitations. It brings many points of view to the table, encourages discourse, and prevents key decisions from being made by only one person in isolation. It is also a way to determine what pertinent safety data are or are not currently available to the team. Finally, it helps ensure that goals, objectives and timelines are understood by all. Haz-Op participants should include those in-house experts who have the greatest familiarity with the process and the facility. The person calling or chairing the meeting should be someone knowledgeable about leading such reviews and who has a grasp of the “big picture” of the project.

Conducting a proper Haz-Op takes time. Making it effective requires that all participants do their homework beforehand. Many hazards are not immediately obvious, and the preventive actions to be taken may involve making choices or changes that affect other areas. Equipment modifications may be governed by change-control rules, or GMP guidelines. Some proposed changes may be very extensive and not justifiable based on the goals of the project. These should be group decisions. Unless it is a very simple project, a complete review will likely take more than one meeting. The bigger the operation and the more potential hazards, then the more people and time will be involved. All new processes deserve a review, even if they are similar to existing processes.

Helpful Documentation – Certain documents are critical to conducting an effective Haz-Op, and preparing some of them can be a time-consuming proposition. The chairperson should see to it that the necessary documentation is prepared by the appropriate parties and distributed to all attendees well in advance of the meeting. The table at the bottom of the page lists some of the items that may be useful for the review.

Agenda – The agenda should also be distributed before the meeting. Possible meeting topics include:

- Introduction: A summary of campaign goals and objectives, projected product needs, timelines and deadlines.
- A review of the chemistry involved and its potential hazards (exothermic reactions, off-gassing, etc.). Review of DSC and calorimetry for such aspects as heat of reaction, thermal runaway, decomposition or polymerization reactions, gas amount and pressure generated.
- The important safety, handling and disposal characteristics of the reagents, solvents and unlisted intermediates involved. Safety, health, and environmental data on any hazardous materials and effluent streams. MSDS salient

Important Documentation for Use During Haz-Op
Laboratory Process Description, in at least draft form, outlining the chemistry and conditions involved, with laboratory yield and quality data.
Process Flow Diagram (PFD, see pg 1-13) to facilitate process review.
Piping and Instrumentation Diagram (P&ID, see pg 2-2) to facilitate review of the equipment train.
Draft batch record to facilitate step-by-step discussion of the proposed operation (see pg 1-14).
Process Safety Information - calorimetry results and other hazards information, including raw material and intermediate MSDSs, materials compatibility issues, secondary containment, waste streams, gas emissions, etc.

points and any other data on safety and health effects.

- A step-by-step review of the process and detailed discussion of how each step, each chemical transfer, and each unit operation will be performed. It is useful to walk through the batch record for this part. It should include a discussion of the proposed equipment, material compatibility issues, and waste disposal plans. Discuss process operating limits – pressure, temperature (including jacket temperature limits), concentration and time limits. Has the process been tested at those limits? Should distillations be carried out under vacuum or at atmospheric pressure? Discuss the sampling plan. What are the criteria for reaction completion and other in-process controls? Discuss equipment selection for isolation and drying. Discuss possible stopping or hold points, especially if 24-hour operation is not planned.
- Review of equipment (P&ID). Discussion of possible equipment changes and modifications and change-controls governing them.
- Special coverage needs for round-the-clock operations, especially in facilities that do not normally operate three shifts.
- Review and sign-off of master production record.

What If? – Throughout the Haz-Op, the chairperson should encourage a “what if?” attitude among participants. The purpose of the Haz-Op is to prevent potential accidents before they occur, and intuition, experience and imagination are needed to anticipate potential mishaps. Questions like “what is the worst possible thing that could go wrong?” should be encouraged, even if they seem farfetched, because such questions can often influence other team members to stretch their imaginations as well. Here are some possible questions and areas to consider:

- Consider what happens during start-up, shutdown and other abnormal conditions.
- Consider the possibility and consequences of equipment failure, along with contingency plans and corrective action. Examples might include loss of agitation, loss of heating or cooling, compressed air failure or power outage.
- Determine the need for backup equipment and spare parts.
- Discuss preparedness for vessel breakage, leaks or spills, and responses to such events.
- Consider possible interactions between vessel contents and heat transfer fluids.
- Consider the possibility of operator error – over charging or undercharging raw materials, adding materials in the wrong order, omitting a component, overheating, holding for too long at reaction temperature, opening or closing the wrong valve, etc.
- Consider safeguards to prevent adding “all-at-once” a reagent that was intended for slow addition.

Obviously it is not possible to identify all potentialities, nor to account for all possible human errors but this approach can go a long way toward raising awareness of possible adverse events, and brings preparedness to the front of everyone’s mind.

The proceedings of Haz-Op meetings should be documented in detail, either in the form of minutes, or a report summarizing the meeting, distributed to all participants and key management to record what was discussed and agreed upon. This report makes it easier to follow up on action items and can help limit liability by demonstrating due diligence on the part of the company.

Starting a Haz-Op program from scratch is not easy and it won’t happen overnight. If Haz-Ops are new to your organization, it is wise to start small and build. Slowly work to counter the idea that it is just another layer of bureaucracy. It is a program that works and pays untold benefits in the long run in process efficiency and worker safety. Getting management buy-in early is important to long-term success. Haz-Ops will eventually be seen as important part of operations and will be accepted as a normal part of process scale-up. Suggested further reading [104, 137].

The Batch Checklist

Careful planning is the lifeblood of a successful scale-up operation. The saying goes that “the devil is in the details” and there are certainly countless numbers of them. The list below includes some of the more important items that the process champion will need to think about when preparing for the all-important pilot run. Many will be carried out in parallel. It is important to fix batch size early so that long-lead materials can be ordered and special equipment or setups can be installed and tested. It is equally important to understand the goals, objectives and purpose of the batch or campaign, including expectations about timelines and the minimum amount of product required. It is also assumed that process safety considerations and specific processing concerns or trouble spots have been discussed by the team at large.

Some Tasks Associated with a Pilot Plant Batch

Raw Materials

Raw material and specifications list complete
All raw materials ordered
RM analytical methods in place
All raw materials received
All materials sampled for release
All materials released
Raw material use test completed
Use test product analysis complete

Misc. Materials/Disposables

Extra solvent for line rinses
Inert gas supply
Filter media
Cartridge filters / Filter aids
Process water system released

Process Safety

Max/min volume steps identified
Critical processing steps identified
Material compatibility confirmed
Reaction calorimetry completed
Safety assessment completed
Haz-Op completed
Ensure adequate cooling capacity
Estimate effluent streams and plan for disposal
Spill response procedures and contingency plans
Notify facilities, security departments of overnight operations

Scheduling

Ensure equipment availability
Schedule sufficient manpower
Contingencies in place for extended operation

Equipment

Equipment train selected:
reactor, dryer, filter, etc...
Instrumentation needs identified
Compatibility of all seals and gaskets checked
Special setups or apparatus complete

Sampling apparatus in place

Equipment cleaned, released
Equipment dry
Equipment leak-tested, pressure-tested
Equipment log books up-to-date
Spare parts on hand

Utilities

Chiller
TCU
Vacuum
Scrubber
Compressed air

Process Documentation

Laboratory Process Description complete
Process Flow Diagram complete
Master Production Record complete, approved
Environmental permitting complete
Batch Record in hand
All MSDS sheets present
Hazardous properties of intermediates identified
Master campaign file opened

In-Process Controls

In-process checks identified
Sampling plan in place
Analytical methods available
Analytical support scheduled
Ready to collect, quench and store samples

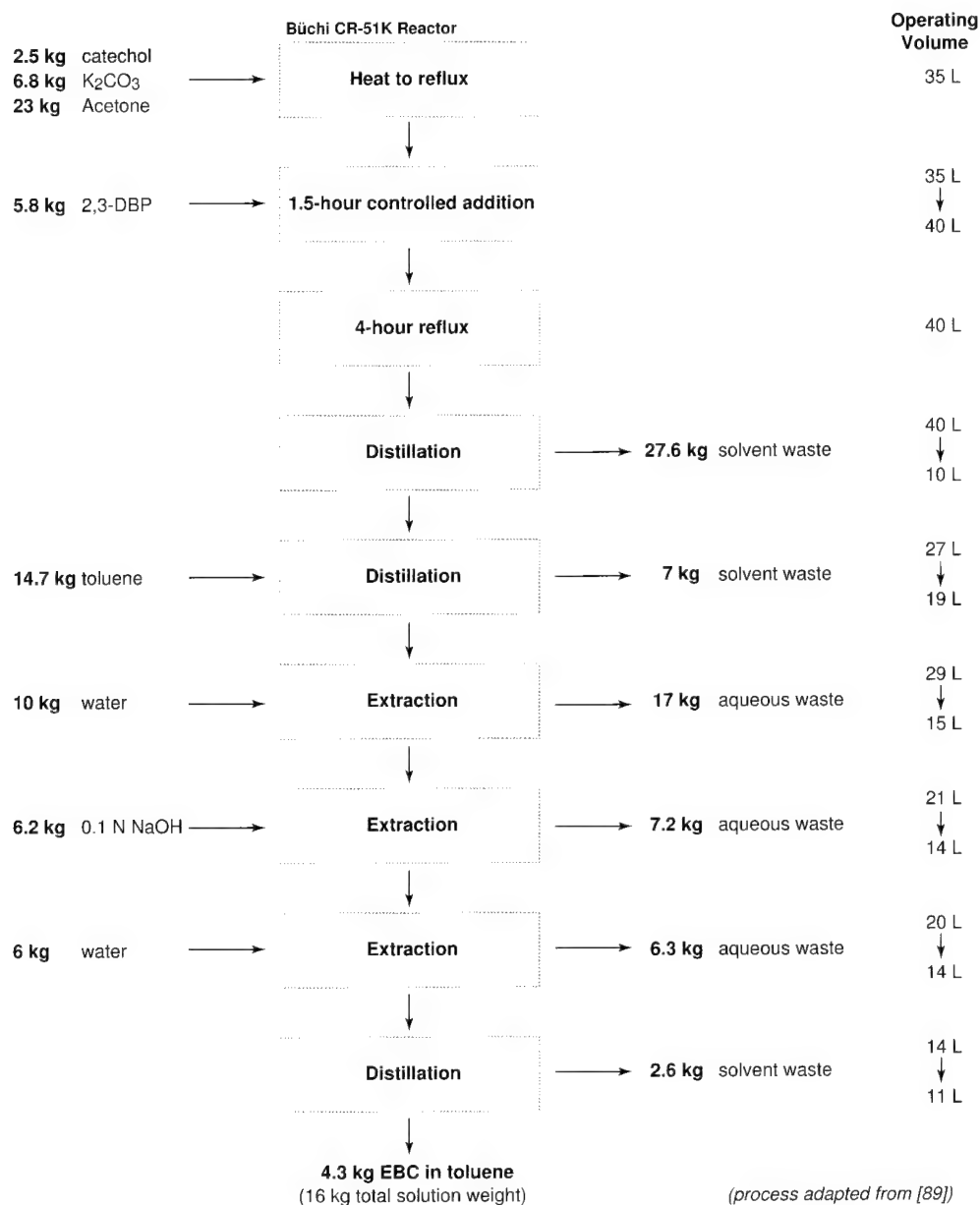
Follow-up

Product packaged and labelled
Retain samples collected
Sample analysis complete
Data analysis complete
Mass and energy balances complete
Batch/campaign report complete
Special apparatus or rigs broken down
Equipment modifications reversed
Equipment cleaned and released

The Process Flow Diagram

The Process Flow Diagram (PFD) is an invaluable tool for conducting reviews and simplifying communication about a process between team members of various disciplines. One possible format is shown below. This PFD was created as a spreadsheet, using the charge of the raw material catechol as the basis. When the charge of catechol is changed, the rest of the values, including the weights of waste streams and operating volumes, are automatically calculated. This is a great time-saver when exploring possible batch sizes and process modifications. It gives an immediate indication of minimum and maximum operating volumes to simplify vessel selection, and allows an at-a-glance determination of raw material needs. This flowsheet is available for downloading as a template from the website www.pprbook.com.

A Typical Process Flow Diagram



The Batch Record

The batch record, sometimes called the “batch ticket” or “production record”, is a blank form or log-sheet on which operators can record all pertinent processing information, such as raw material lot numbers, weights, temperatures and pressures at key steps, reaction times, etc. It becomes a permanent record of the batch for inclusion in the campaign file. More often than not it is a paper hardcopy, although some companies are moving toward electronic batch records. Usually, especially in GMP facilities, the batch record is an official numbered photocopy of something called the Master Production Record (MPR) which is an original hardcopy reviewed and signed by representatives of various departments including R&D, Engineering, Manufacturing, QC, QA, and other project managers. This provides a way to keep track of revisions and ensure that operators are always using the most up-to-date version. Even in non-GMP environments, change control of this nature is extremely important.

Using a batch record is a good idea no matter how large or small your operation. It provides a means of ensuring that steps proceed in the correct sequence, that no step is overlooked, that operating ranges are kept within the specified limits, and that no important data are lost. Many possible formats exist, any one of which is fine, as long as it includes the necessary sections. An example of a typical batch record, which can be used as a template, is available for downloading from the website www.pprbook.com.

Below is a list of some of the sections that should be included in a batch record:

- **Title** – process name, product name or material code number, as appropriate.
- **Revision Number and Effective Date**
- **Approval Signatures** – including author, development chemist, and project manager.
- **Brief Process Description** – including reaction scheme and a balanced chemical equation. Note critical processing steps and conditions.
- **Process Flow Diagram** – see page 1-13.
- **Raw Materials List** – including synonyms, code numbers, quality specifications and possibly a list of suppliers.
- **Equipment List** – indicating precisely which equipment will be used for the various processing steps.
- **Safety Data** – key safety information on hazardous substances used in the batch, special warnings, recommended personal protective equipment (glove material, respirator type, etc.), spill control and cleanup procedures.
- **Raw Materials Calculation Table** – for determining the amount of each component used in the batch. Calculations are usually based on the charge of a single key raw material, equipment size, or some other fixed basis. Raw material ranges and expected yield should be included.
- **Batch Size Limits** – minimum and maximum batch size that can be operated in the specified equipment.
- **Processing Section** – step by step processing instructions with blank spaces for recording:
 - Starting and ending time for each step (military – or 24-hour – time is less ambiguous)
 - Operator initial's for each step, countersigned by second person for raw material charges and other key steps
 - Raw material lot numbers or inventory control numbers
 - Raw material charges (gross, tare and net weights)
 - Tables for recording controlled reagent additions over time
 - Batch temperature, pH, pressure or other applicable parameters
 - Sampling – type of sample, time of sample, sample ID number, etc.
 - Intermediate stream or effluent stream weights
 - Drying record
 - Product wet, dry and packaged weight
 - Collection of samples for analysis and retain
- **Yield Calculation Page** – indicating % of theoretical yield and % of expected yield.
- **Batch Closeout** – signatures to ensure that all sections are complete and all reports and attachments are present.

The Campaign Report

Pilot batches provide unique opportunities to collect data and information on process performance that cannot be obtained any other way. The pilot campaign will prove even more valuable in the long term if the observations, results and analytical data are gathered together in a single place where they will be easily accessible later on. An important part of this is data reduction – tabulating the key results in ways that are clear and that make comparisons easier. The campaign report is the ideal repository for all of this information. It's a good idea to generate a standard format for the report to ensure that all key information is included and that differences in writing style will not result in important data being excluded.

Summarizing the findings and committing the results to paper will prove very beneficial in critiquing process performance and helping to identify areas for optimization or further development work. Troubleshooting is also easier.

Here is a short list of important things that should be included in the campaign report:

Summary Table – List all batches, dates, yields, key analytical results and purity information, to save time and simplify comparisons between campaigns that used different processes or were carried out at different facilities.

Batch-by-Batch Process Summary – Here list the detailed operations, actual raw material lots and charges, processing times and operating conditions for each batch. This will make it easier to make batch-to-batch comparisons and tabulate critical process parameters and results when necessary later on.

Materials List – Include all raw materials and intermediate sources, lot numbers, important quality characteristics, etc. Include copies of manufacturers' certificates of analysis (COAs) for future reference.

In-Process Control Results – Include all process checks including results of sample analyses. Attach analytical data sheets. A table comparing these results to those obtained in bench work should be included.

Analytical Results – Tabulate the results of all product analyses, including averages, standard deviations and expected (bench) results for comparison. Include Certificate of Analysis as well as raw data, chromatograms and other quality reports. Other attributes that may not be a part of the product specification, such as particle size analysis, should be included as well. This will simplify assessment of the scale-up operation. Make sure to complete the comparison with material prepared on the bench for every quality attribute, and work to find reasonable explanations for any differences. Note that the impurity profile can be affected significantly by extended operating times, operating outside of the specified limits or by possible non-homogeneous conditions in large-scale equipment.

Mass Balance – Two types of mass balance assessments can be valuable. The purpose of the *overall mass balance* is to determine if the mass of all of the materials that went into the batch equals the mass of the materials that came out. This helps identify losses due to solvent evaporation, gas emissions or handling issues. Scrub liquors and waste streams must be accounted for. The *key raw material mass balance* seeks to account for all of the major building blocks of the product molecule on a molar basis. Such a study requires careful measurement of all input streams as well as quantitative analysis of all waste streams, mother liquors, cake washes, cleaning solutions, scrub liquors, liquid from drying traps, etc. Much more is learned from this study than from an overall mass balance, in terms of recoverable yield and reaction performance at scale. Investigations into yield discrepancies must start here. It is a critical exercise in commercialization of any chemical process.

Waste Stream Report – List the weights or volumes of all waste streams for disposal as hazardous waste. This must include all extraction streams, equipment cleaning solutions, mother liquors and washes, scrub liquors, and solid waste from desiccation or decolorization operations. Calculate the amount of each waste generated per unit mass of product.

Energy Balances – In situations such as exothermic reaction control or temperature ramping during crystallizations, it is important to recognize any unexplained temperature excursions or heat transfer steps that took longer than expected.

Attachments – Include copies of all batch records, drying records, deviation reports, analytical reports, certificates of analysis, etc. Depending on the situation, the original batch records may need to be archived elsewhere.

Notes on GMP

Good Manufacturing Practice (GMP) is a detailed set of guidelines and procedures used by pharmaceutical manufacturers to ensure that they can consistently make active pharmaceutical product (API) that meets its purported quality specifications. It is sometimes called “cGMP” for *current* GMP to reflect its state of ongoing revision. It represents an over-arching operating philosophy, and while its existence is legislated by law (21 CFR 210 and 211), its details are not. Thus there is considerable flexibility in how the goals of GMP are accomplished. To help meet FDA requirements, the industry has adopted a set of guidelines established by the Pharmaceutical Manufacturers Association (PMA). These guidelines advise manufacturers on the many aspects of drug development, testing, manufacture, quality assurance, quality control and documentation.

It is not possible to present all the details of the PMA guidelines here, but a brief listing of the key areas regulated by GMP may be useful. Note that written, approved Standard Operating Procedures (SOPs) must be in place for all operations, no matter how minor, in order to help ensure consistency and minimize differences in approach between personnel. In general, complete documentation must show that the following systems are in place.

Personnel and Training – All personnel involved in the manufacture, testing, packaging and labeling of drug products have the education, experience, qualifications and training to perform their functions and follow applicable SOPs; personnel training program with regularly scheduled sessions and training records retention system are in place.

Buildings and Facilities – Buildings and facilities designed and qualified for the manufacture of drug substances; layout minimizes cross contamination and mix-ups; air handling and dust control systems, temperature and humidity controls, microbial and pest control systems, sanitation and housekeeping programs are in place.

Research, Development and Scale-Up – Experimental design, data collection, notebooks and reports support the selection of the process; all major process changes are supported by a reasonable rationale; process control implementation program is justified; critical process parameters and quality attributes are identified; data demonstrate equivalence in purity at various scales.

Material Control – Procedures for ordering, receiving, labeling, quarantine, sampling, testing, release, storage, expiration dating and disposition of all raw materials, product, and other items used in the process; suppliers are qualified.

Equipment Qualification – Documentation of completed Installation and Operational Qualification; procedures for equipment change control, maintenance and repair; validated cleaning protocols and results; Instrument calibration schedule in place and calibration records available.

Manufacture Control – Approved batch records and procedures for documenting deviations; in-process testing, including validated test methods and criteria for acceptable results; procedures for investigating and documenting failed batches and yield discrepancies; justification for reprocessing and 2nd-crops; computerized control systems validated.

Quality Control – Validated analytical test methods; system suitability tests, determination of sensitivity and limits of detection; primary and secondary reference standards established; sample retention; stability testing program, including accelerated stability testing, forced degradation studies, isolation and characterization of degradation products.

Raw Material and Intermediate Specifications – Sampling protocols; establishment of criteria for identity, purity and strength with validated test methods; supplier approval; release and reject procedures; expiration dating system; testing schedule, sampling methods and quality standards for purified water systems.

Product Specifications – Written rationale for all specifications; identification of relevant physical characteristics, including crystalline form, impurities, homogeneity and microbial contamination; stability studies; expiration dating; labeling; validated analytical and test methods; identification and characterization of major impurities.

Process Validation – Approved validation protocols for all key processing steps (the process does what is purported); raw materials and conditions clearly defined; sources of process variation identified and minimized; key process parameters monitored and challenged to ensure conformance to specifications; effect of operating condition changes evaluated; repeat validation batches completed; revalidation performed if there are significant process changes.

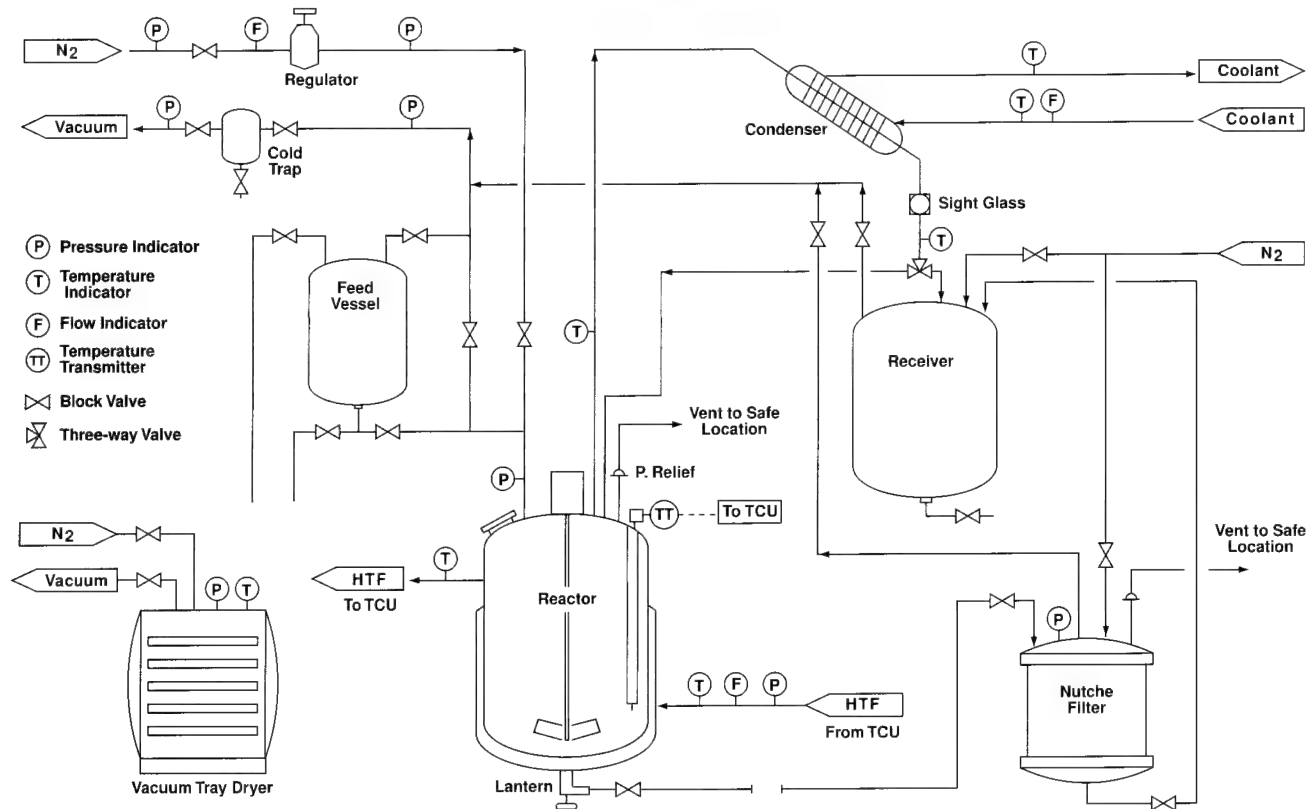
Documentation – SOP and production record approvals; revision and change control; manufacturing records, deviation reports, training records, etc. archived.

2 Equipment and Operations

Contents

The Pilot Plant Equipment Train	2-2
Notes on Equipment Selection, Installation and Maintenance	2-3
Reactors	2-4
Vessel Agitation	2-8
Charging Raw Materials	2-11
Reaction Control	2-13
Sampling for Reaction Monitoring	2-15
Workup	2-16
Partition Coefficient and Extraction Efficiency	2-17
Polish Filtration / Decolorization / Water Removal	2-18
Batch Distillation	2-19
Crystallization	2-21
Product Isolation	2-24
Filtration Scale-Up	2-27
Product Drying	2-29
Scrubbers	2-33
Reactor and Equipment Cleaning	2-34

The Pilot Plant Equipment Train



The figure above shows a simplified Piping and Instrumentation Diagram (P&ID) for a typical batch reactor equipment train. Jacketed reactors are usually coupled with a feed vessel for liquid addition, a receiver for collecting distillate and liquors from product isolations, a filter or a centrifuge and some sort of overhead condenser. Utilities normally include vacuum (with cold-trap), nitrogen for inerting (which must be available at all vessels), coolant (usually cold water or glycol) to feed the condenser, and a temperature control unit (TCU) or other source of heat transfer fluid (HTF) to control reactor temperature.

The reactor should always be fitted with a lantern (double-valved sight-glass) for making phase splits, pressure indication and overpressure relief (such as a rupture disk) vented to a safe location, and temperature indication and control. Temperature and flow indicators on the condenser cooling loop, vapor and condensate lines and on the jacket loop are valuable for closing energy balances, determining heat transfer coefficients, and troubleshooting.

Not shown on this diagram, but highly recommended, are a scrubber for neutralizing noxious gases, a Dean-Stark apparatus for removal of water during condensations and other dehydration reactions, a torque meter or watt meter for determining mixing power draw, a sample collection apparatus (see page 2-15), an automatic vacuum control valve, reactor volume calibration or level sensor, a metering pump for controlled additions, and a temperature recorder or data logger to follow trends in real time.

The following sections of this chapter discuss the various types of equipment found in a typical kilo-lab or small pilot plant in more detail, including suggestions for its selection, care and operation. The sections are arranged in the roughly chronological order of a typical synthetic chemical batch sequence. It is difficult to separate discussions of the equipment and its use, and therefore characteristics of the units themselves and recommendations for their safe and effective operation are often intertwined in the text. Tips, pointers, and tricks of the trade from many sources, including the personal experience of the author, are offered in an effort to educate the reader, and hopefully, to generate some new ideas among experienced tech-transfer staff.

Notes on Equipment Selection, Installation and Maintenance

Selecting the proper equipment, no matter how large or small, should be based on your projected needs, budgetary constraints, and in close cooperation with your vendor. Any reputable supplier will help you understand how the equipment will perform for you and should be willing to advise you every step of the way. They are most familiar with their equipment and usually know exactly what it will or will not do, and know the required ancillary equipment and utilities. Competition is stiff. Check out as many alternate manufacturers as practical. Choose a vendor who is reputable, with whom you feel comfortable, and who is willing to spend time with you to ensure that you are satisfied. The internet, trade shows, industry periodicals, and your own colleagues are all good starting points for locating equipment.

New or Used? – Don't overlook the real bargains available in used processing equipment. The chemical industry is a tumultuous place. Companies come and go, and many pieces of equipment labeled "used" have hardly been used at all, if ever. Obviously, it is important to obtain the full history of the item, and have it inspected by someone knowledgeable in the field. Even in GMP facilities, purchasing used equipment is not out of the question, but it is much more difficult since the history must be documented in detail, including IQ/OQ (see below), maintenance and cleaning records. Aaron Equipment Co. and Manufacturer's Mart are two well-established sources of used equipment.

Installation – Because of the many potential hazards inherent in chemical processing, equipment installation should be undertaken only by qualified parties. Many units have powerful moving parts, and a proper support structure must be provided to prevent vibration and failure. Electrical motors will most likely have to be explosion-proof rated (see page 5-12), and the wiring must be performed by a certified electrician. Very often installation assistance can be provided by the vendor, or the vendor can recommend certified contractors, if necessary.

IQ/OQ – New equipment installed in a facility must, of course, be tested to ensure that it is working correctly. However, given the complexity of processing equipment, "working correctly" may not be so easily defined. For this reason, it is standard practice (and certainly *required* practice in GMP environments) to perform a comprehensive series of tests called Installation Qualification and Operational Qualification (IQ/OQ). The tests must follow a carefully-planned protocol and the results become part of a permanent record for that unit. At a minimum, even in small operations, the results of IQ/OQ should be recorded in the equipment log book.

IQ ensures that the equipment has been properly installed and operates as advertised by the manufacturer. Does the agitator turn at the right speed and in the right direction? Does the system hold vacuum without leaking? Does the controller properly maintain setpoint?

OQ on the other hand is designed to answer the somewhat trickier question of whether the equipment will do for you, the user, what you expect it to do. Will the TCU heat the reactor full of water to 90°C in one hour? Can the system distill your solvent at the required rate of 50 liters/hr? Does the agitator mix your biphasic reaction with sufficient turbulence? While IQ criteria can be worked out together with the manufacturer, OQ tests are the purview of the equipment user, and can be quite involved. It is sometime desirable to run tests that mimic batch operations without charging actual reagents or solvents, and it can prove difficult. Ultimately the goal is to demonstrate, to everyone's satisfaction, that the equipment will work for whatever purposes you eventually have planned for it. At any rate, running solvent through a new equipment train should be *de rigueur* before ever introducing reagents.

Maintenance – Once a piece of equipment is installed it must be maintained in strict accordance with the manufacturer's recommendations. Someone must be responsible for scheduling this and seeing that it happens. A comprehensive preventive maintenance (PM) program should be in place for all lab or plant equipment. Keep permanent records of any equipment maintenance, repairs and modifications, be it as simple as changing the vacuum pump oil, or replacing a gasket. Not only is this good engineering practice but it is also required by the FDA for cGMP facilities.

Spare Parts – As part of the PM program, it's a good idea to keep a stock supply of the most common wear items and spare parts – gaskets, O-rings, fuses, bulbs, etc., for your equipment. This will minimize downtime and increase efficiency. But equally important is *knowing* what parts are on hand and being able to find them, which is why a good parts inventory and storage system is needed. Manufacturers and vendors will help you determine the most useful spare parts to have on hand. Keeping a list of their part numbers and supplier contact information will help speed replacement.

Reactors

The reactor is the heart of any chemical operation, and, not surprisingly, a great many reactor styles, sizes, configurations, and materials of construction are offered by many manufacturers. The purpose of the reactor will, of course, determine its design. Batch reactors, cascaded batch reactors, continuous reactors, tubular plug-flow reactors, spray reactors, falling film reactors, and others all serve specific functions and are common in the industry.

However, in the pharmaceutical and fine chemical industries, where low-volume, high-value products must be made in multiuse equipment, the **batch stirred-tank reactor** is the most common. It represents the lowest capital cost and provides the greatest flexibility among reactor types. As mentioned previously, the batch operating mode is perhaps the most labor intensive and results in the most downtime, but it also allows the quickest process scale-up. Because of its widespread use, the discussion here will be limited to this class of reactors.

Most vessels used as reactors are cylindrical with a centrally-positioned vertical mixing shaft. Ends are usually dished to accommodate pressure or vacuum operation. Height-to-diameter ratios between 1 and 1.5 are typical. Heat transfer area can be increased by making the vessel taller, but bulk mixing may suffer. Baffles are usually installed near the vessel inner wall to prevent “swirling” and improve mixing efficiency. Baffles can also house temperature sensors, pH probes, or act as dip tubes for gas injection or sample collection. Internal cooling coils are sometimes added to improve heat transfer.

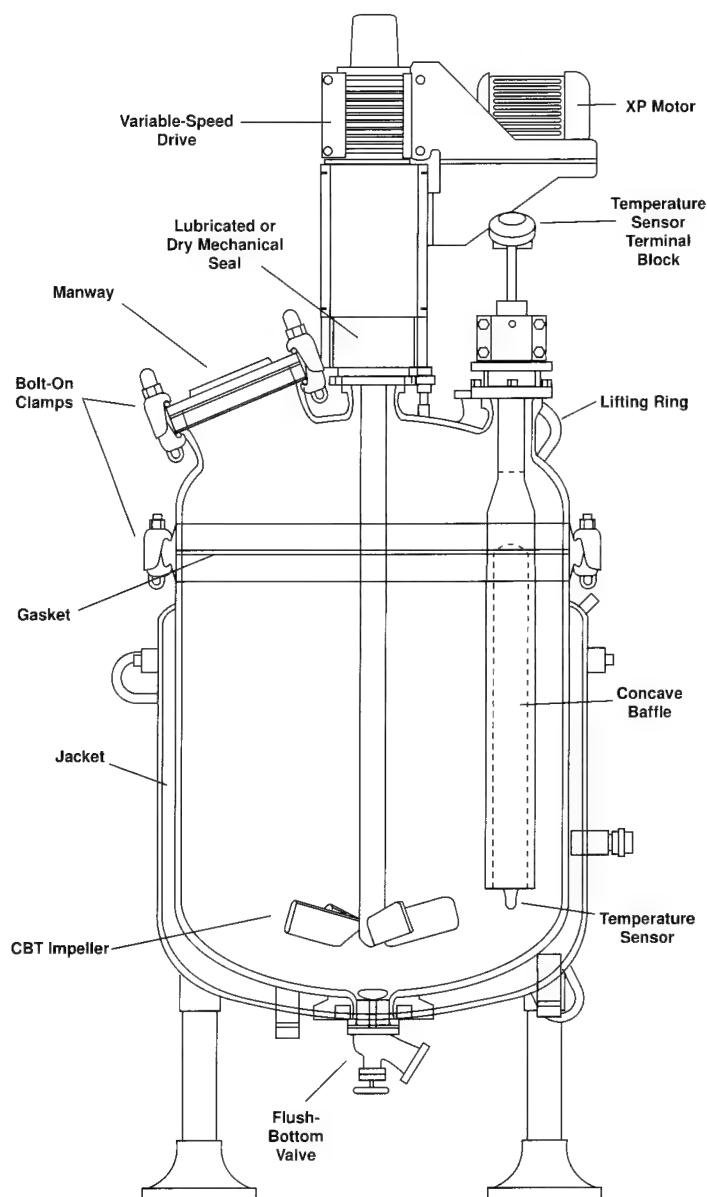
Material of Construction –The first and most common choice for reactor material is glass, because of its excellent chemical resistance. It is also smooth and relatively easy to clean. Small vessels may be made entirely of glass, and for larger systems, glass-lined steel units are quite common. These are steel vessels that are coated on the inside with a layer of high-temperature fired glass enamel about 1-2mm thick. This offers a vessel with a smooth, cleanable, chemical resistant glass surface with great mechanical strength. Pfaudler is considered by many to set the standard in glass-lined steel reaction vessels. They manufacture vessels coated with a number of special glasses, in sizes ranging from 10-gal to 4000-gal, which can operate at internal pressures in excess of 100 psig. An example is shown on the following page. De Dietrich is another highly recognized manufacturer of excellent glassed-steel equipment, as is Tycon Technologies.

Glass-lined reactors are often manufactured in two sections. The sections are usually joined and sealed by using a PTFE-envelope gasket and bolt-on clamps positioned every few inches around the entire perimeter. This same type of clamp is used to secure the manway, a large hatched opening used for solids charging, agitator replacement, and inspections.

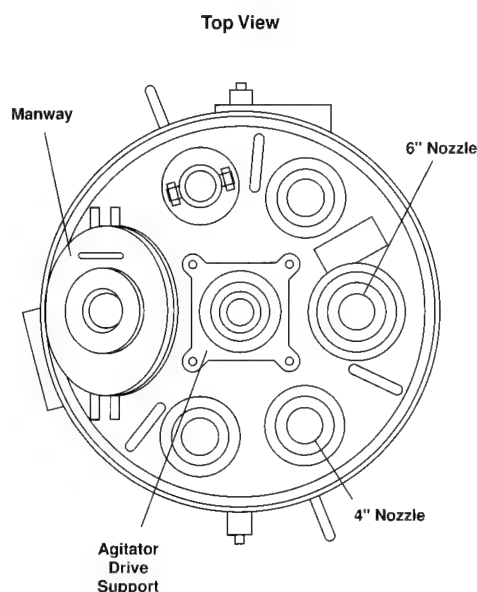
Visibility in glass-lined tanks is limited to a small sight glass and this is often clouded by condensation (a brief nitrogen sweep through the reactor can help clear this). For smaller pilot equipment, up to, say, 150 liters, some may prefer the full visibility offered by a solid glass reactor system. It is often valuable to be able to observe the performance of certain operations. It is the author's opinion that there are no finer systems than the Swiss-manufactured Büchi line, such as the one shown on page 2-6. This is actually a hybrid system consisting of a transparent upper portion and a glassed-steel lower portion, which allows higher jacket pressures. Many simpler, but much less versatile, blown glass systems are available, including those offered by companies such as Martin Glass (see page 2-6), Ace Glass, Schott Glass, and Chem-Flowtronics.

In spite of its obvious advantages, glass does have limitations. First, it is breakable. It also has a lower thermal conductivity and thus poorer heat transfer than metal reactors. It cannot be used at extremely high or extremely low temperatures, and cannot be exposed to large temperature differentials. Carefully consider the ΔT when adding media to a reactor or the jacket. The common rule is a maximum ΔT of about 50°C for glass-lined steel and about 100°C for solid glass systems. In other words, never add cold product to a hot reactor, or inject cold HTF into the jacket of a hot reactor. Doing so could shock the glass, causing it to crack or separate from its metal substrate. Also, glass cannot be used with hot alkali and similar substances, and has definite pressure limitations. More on the chemical resistance of glass can be found in Chapter 10.

When it is necessary to circumvent these limitations, metal reactor vessels are often employed. The selection of metal depends on the application, but 316 stainless steel is the most common. It is durable, has excellent heat transfer characteristics and can be manufactured to meet nearly any temperature or pressure requirement. This makes it best for highly exothermic reactions, hydrogenations, and for very low temperature work. Where greater resistance to acids is required,



**A Contemporary
200 Gal Glass-Lined
Steel Reactor**



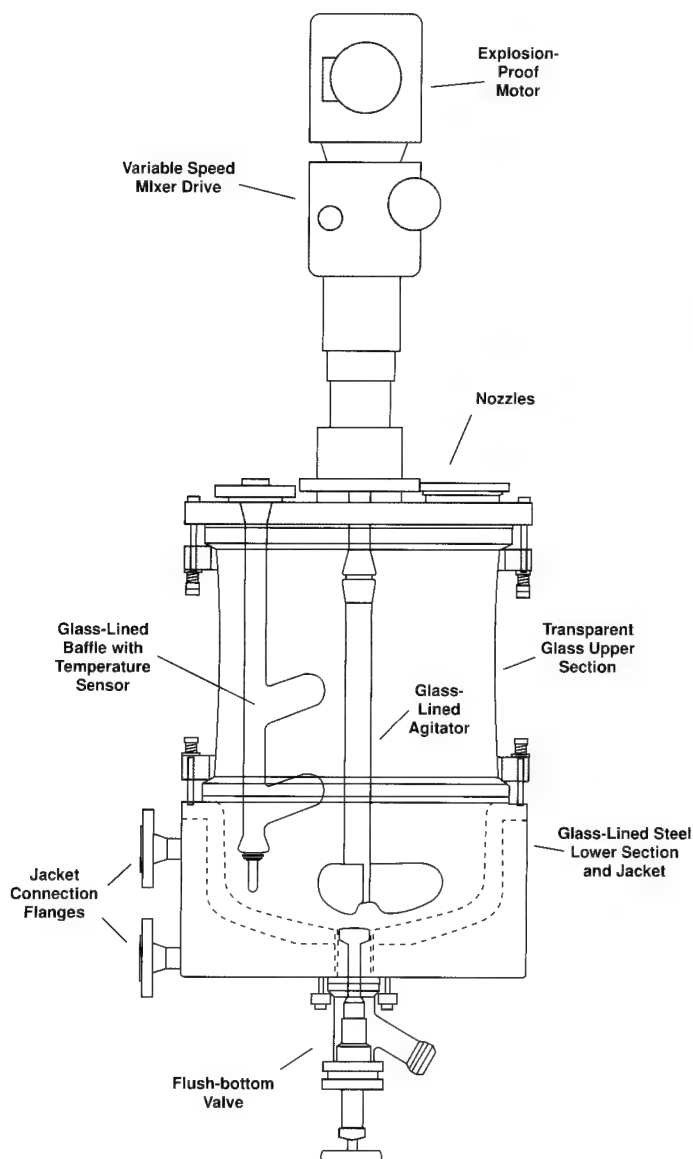
Adapted with permission, Plaudler, Inc.

Hastelloy is the next common choice. This is a very expensive, but highly chemically resistant, nickel alloy. For more information on the properties of common metals and alloys see page 10-5. PTFE-lined vessels are also an option, but heavy PTFE linings can reduce heat transfer performance.

Design Specifics – Writing a detailed reactor specification requires knowing the expected use and operating mode. As mentioned above, this discussion is limited to stirred batch reactors. Multiuse vessels are usually selected from standard sizes and models, but it is always possible to customize them to meet special requirements such as agitator type and speed, heat transfer surface area, type of jacket, material of construction, etc.

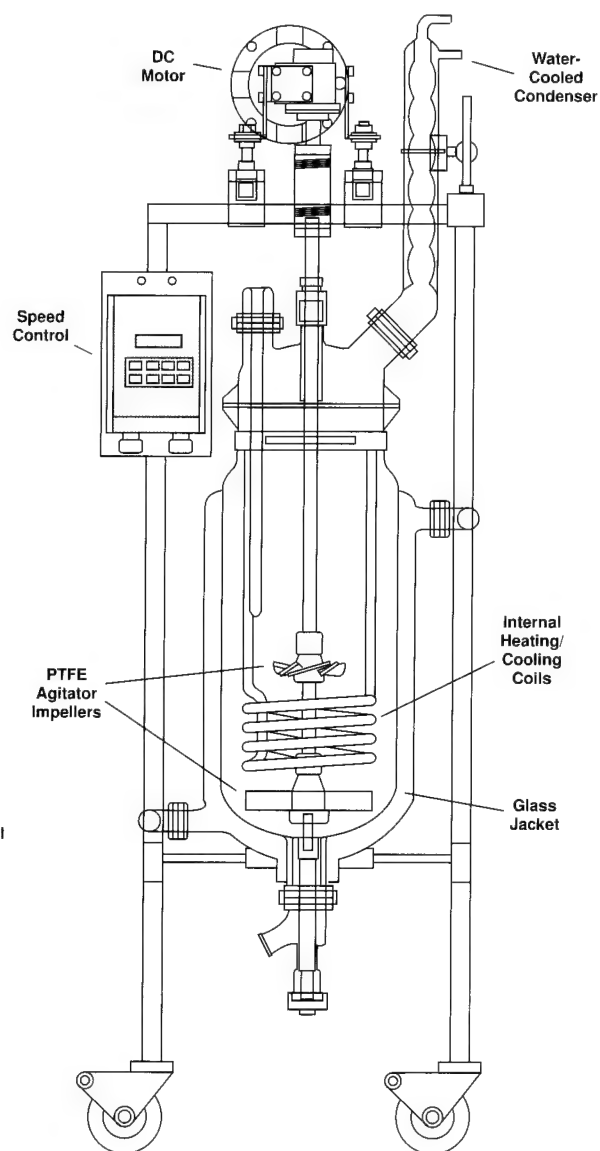
Size – Start by considering your volume needs. Most standard reactors are designated by nominal volumes, but determine the actual maximum capacity and minimum mixing volume before purchasing. Watch the distinction between U.S.

2 - Equipment and Operations



70-Liter Glass Reactor

Adapted with permission, Büchi-Glasuster



Portable 50-Liter Glass Reactor System

Adapted with permission, Martin Glass

gallons and U.K. (or Imperial) gallons. For a typical 200 gallon (US) glass-lined reactor, the maximum volume with full agitation is ~220 gal; the minimum volume for effective mixing (at medium agitation) is ~20 gal.

Construction – Next examine your chemical compatibility requirements and expected temperature and pressure operating ranges. Make sure there are enough nozzles for the required connections – distillation condenser, reflux return, feed lines, scrubber, pressure gauges, vacuum, nitrogen, overpressure relief, pH and temperature probes, dip tube for sampling or gas injection, spray ball, etc. For glass-lined tanks, include a window-type sight glass, or two if possible, one for lighting and one for viewing. Low-profile explosion-proof fiber optic lights are useful accessories.

Pressure rating – The safety rating of all pressure vessels is carried out according to the ASME Boiler and Pressure Vessel Code. Make sure that any vessel intended for use above atmospheric pressure is ASME rated. Overpressure

protection must be provided. This can take the form of a pressure relief valve, but a rupture disk is usually standard on all larger vessels. This is a calibrated disk, usually made of special alloys or graphite, that will burst above a certain pressure. Rupture disks are also available with electronic sensors to signal the process controller or sound an alarm if the disk bursts. See the diagram below. Rupture disk outlets should be piped to a catch basin or other safe location.

Agitator – Consider the best agitator type for your needs (see page 2-8), or select a system with easily-changed impellers. Variable speed mixing is best if available. Unless you indicate otherwise, manufacturers usually supply a standard motor horsepower size for each of their vessels (page 2-8). The mixing shaft seal is a critical component of the stirring mechanism, especially for pressure or vacuum operation. There are a wide variety of rotary seal types, including wet seals in which a thin film of water or glycerin is used to lubricate the shaft seal elements and complete the seal. Water-wet seals should be avoided if possible, since they have been known to leak into the batch. There are many excellent ceramic, gas pressurized, and other dry seals that are dependable, low maintenance and rated for pressure applications.

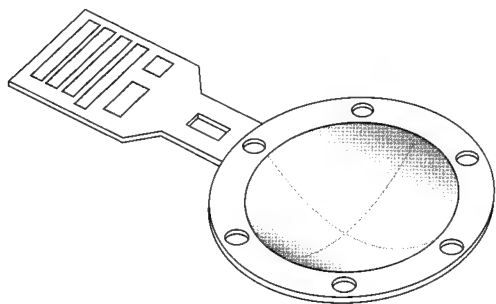
Other – The bottom drain valve should be of a type that will not clog with solids. A flush-bottom valve meets this criteria quite well and is standard on glass-lined reactors. A lantern, or double-valved sight-glass, should be installed to simplify phase-splits during extractions. Internal level calibration is offered in some cases. This is usually in the form of a linear numerical scale on the inner vessel wall, which must be calibrated to actual volumes by the user.

Reactor services – Reactor services may include steam, hot water, cold water, brine or some other heat transfer system. For the greatest flexibility and simplicity at the small pilot scale, a separate TCU (temperature control unit) circulating a secondary HTF is highly recommended (see Chapter 4). Liquid nitrogen can be used down to -100°C in stainless vessels, but check with the manufacturer. In-line sight glasses are useful at the point where liquids are charged – there is sometimes no simple substitute for just seeing the liquid enter the tank.

Installation – The location of the reactor and its supporting structure must be considered carefully. Smaller units usually include a supporting framework, which can be installed by the supplier if desired. Such systems can be entirely located within walk-in fume hoods to minimize operator exposure. The installation of larger vessels must be discussed in detail with the manufacturer or other qualified engineering firm. Consider the addition of secondary containment, especially for glass vessels, large enough to hold the entire vessel contents in case of breakage. This can be in the form of a shallow stainless tray, an in-floor catch basin or a sump that can be pumped to an empty holding vessel.

IQ and OQ – The purposes of Installation and Operation Qualification are discussed on page 2-3. In addition to the standard tests suggested by the manufacturer, and those operational tests designed by the user, it is strongly suggested that the following information also be gathered during reactor IQ/OQ. Perform a volume calibration, based on the internal scale, external markings, a calibrated dipstick, or internal landmarks (bottom of the baffle, top of the agitator, etc.). Take the time to calibrate the mixer speed (usually graduated from 0 to 100%) in actual rpm. If possible, measure your impeller power number, N_p , by measuring power draw at different speeds and volumes (see page 2-9). Take the time to measure the overall heat transfer coefficient at several volumes and under different heating or cooling conditions (see page 4-7). Determine maximum heating and cooling rates. These data will prove very useful in future scale-up calculations.

Maintenance and testing – Preventive maintenance for most reactors will focus on periodic inspection and servicing of moving parts such as agitator drives, automatic control valves, seals, gaskets, valve packings, and the like. Instruments and gauges must be periodically calibrated. Follow the manufacturer's recommended maintenance schedule faithfully. Keep a well-organized inventory of spare parts.



A Typical Rupture Disk

Glass-lined equipment has a number of unique requirements because of the susceptibility of the glass lining to cracks and mechanical damage. In order to ensure the integrity of the lining, a **spark test** is performed by the manufacturer. This method uses electrical conductivity to locate possible pinholes or cracks. Glass linings can be patched with tantalum or similar materials when necessary. Another option is to have the entire vessel removed and re-glassed at the manufacturer's site. This is expensive, but much more economical than purchasing a new vessel.

Vessel Agitation

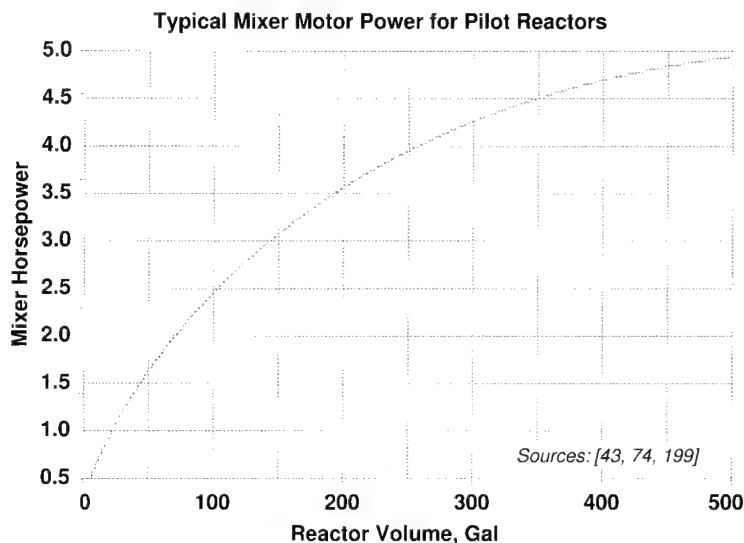
Vessel agitation plays a critical role in heat transfer, mass transfer in multi-phase systems, solids suspension during crystallizations and heterogeneous reactions, and in maintaining homogeneity during chemical reactions. The amount of power consumed by a vessel agitator is a function of the vessel size, the liquid properties and the impeller type. The chart at the bottom of the page shows a rough correlation between vessel size and standard agitator motor size for typical chemical reaction vessels, based on data from a number of manufacturers.

Impeller Design – Many types of impellers have been developed, each with characteristics that make it optimal for certain mixing applications. The traditional impeller design used in the chemical process industry has been the retreat-curve impeller (RCI), also called the “crowfoot”. Originally designed for the polymer industry, it is found in most glass-lined steel reactors manufactured over the last few decades. This blade is not particularly well-suited for all applications, and in fact, suffers from a rather low power number (see below), but it is easy to manufacture and the shape puts less stress on the glass coating. Modern glass technology has expanded the options available for impellers in glass-lined reaction equipment, and now the curved-blade turbine, such as the Cryo-Lock® type manufactured by Pfaudler, is becoming the new general purpose standard.

Other impeller types include the flat-blade turbine, especially good for gas dispersion and emulsification, the pitched-blade turbine, well-suited for solids suspension, and the anchor-style, which is used to improve heat transfer for high viscosity liquids. Most impeller types are available in a wide variety of materials including stainless steel, Hastelloy, and glass-lined. See the chart of impeller designs and characteristics on the opposite page. Many mixing problems are handled by using a multitiered impeller, with two or more impellers, perhaps of different design, mounted on the same shaft. For example, vessels with high height/diameter ratios may provide more area for heat transfer but usually require additional impellers on the shaft. Even then, due to compartmentalization, overall top-to-bottom mixing may be poor.

Scale-Up and Scale-Down – Mixing effects have been studied for decades, and the science of computational flow dynamics has recently been developed to better understand the factors affecting mixing. But mixing is such a complex phenomenon that obtaining the desired mixing characteristics upon scale-up often remains a very empirical exercise. Although some software programs may be of value in experimental design, none can yet eliminate the need for carrying out well-planned mixing experiments.

It is sometimes valuable to be able to “scale-down” mixing conditions, that is, to duplicate at the bench the conditions that will exist in plant equipment. Let’s say a certain reactor is already earmarked for a campaign, and that its important mixing characteristics are known. *Scale-down* experiments may then be performed, in which parameters such as mixing speed, impeller geometry, etc. are varied in an effort to obtain the same mixing characteristics at the small scale. Ideally, an agitation-dependent process developed at the bench using those conditions should perform identically at scale.

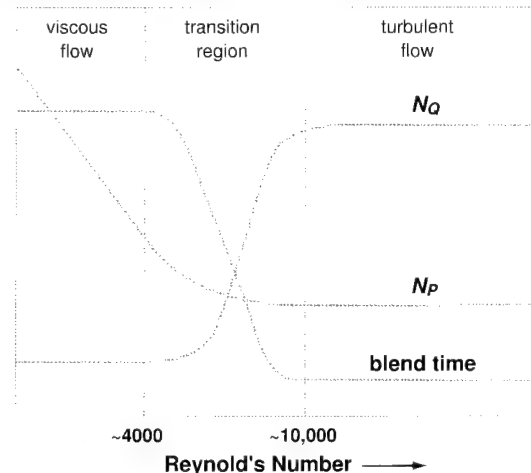


There are three principle ways to consider the scale-up (or scale-down) of physical processes like mixing – by maintaining *geometric similarity*, in which shapes and size ratios only are held equal, *kinematic similarity* in which the ratios of velocities at corresponding points are held equal, or *dynamic similarity*, in which the ratios of forces at corresponding points are held equal. Maintaining dynamic similarity is definitely the most effective when comparing mixing conditions at a smaller scale to those at a larger scale. It enables one to keep the same power input per unit volume, the same fluid “pumping” energy per unit volume, reproduce the same degree of turbulence, or achieve the same blending times.

Useful Relationships for Theoretical Mixing Calculations

Name	Symbol	Function	Units
Reynold's Number	N_{Re}	$= \frac{D^2 N \rho}{\mu}$	dimensionless
Power Number	N_P	$= \frac{P g_c}{\rho N^3 D^5}$	dimensionless
Flow Number	N_Q	$= \frac{Q}{N D^3}$	dimensionless
Energy Dissipation	E_i	$= \frac{N_P N^3 D^5}{V}$	watts/kg
Specific Flow	Q/V	$= \frac{N_Q N D^2}{V^2}$	sec ⁻¹
Tip Speed	V_t	$= \pi D N$	meters/sec

Where D=impeller diameter (m), N=impeller rotational speed (sec⁻¹), ρ =density (kg/m³), μ =viscosity (kg/m-sec), P=mixer power (watts), g_c =gravitational constant, Q=fluid flow (m³/sec), and V=fluid volume (m³). English units can be used as well, as long as they are used consistently.

Dependence of N_P , N_Q , and blend time on N_{Re} 

This diagram shows that although N_P , N_Q , and blend time are functions of N_{Re} , in the turbulent regime they are essentially constant. Adapted from [56].

Mixing Parameters – How well a vessel is mixed is a function of a number of factors: impeller dimensions and speed, vessel size, fluid viscosity and density, and impeller shape and location, to name a few. The classical approach to studying the interaction between these parameters uses a mathematical technique called *dimensional analysis* commonly seen in the engineering sciences. In brief, this analysis allows many complex interactions to be expressed in just a handful of key parameters called *dimensionless groups*. The mixing **Reynold's number**, N_{Re} , is one such dimensionless group. The value of N_{Re} gives a good indication of the nature of the fluid movement in the vessel; values above 10,000 are indicative of turbulent flow, while values less than about 4000 indicate “viscous” (non-turbulent) flow. One way to correlate a mixing process at different scales is to adjust impeller dimensions, mixing speed, etc. to obtain identical values of N_{Re} . While this comparison alone is often not sufficient, it can be very useful. In most large-scale vessels, N_{Re} is usually well above 10,000.

Another key dimensionless group is called the **power number**, N_P . This is a function of power input and is also strongly

Typical Characteristics of Some Impeller Types

Marine-Type Propeller $N_P = 0.8$ $N_Q = 0.5$	Flat-Blade Turbine $N_P = 5.0$ $N_Q = 0.7$	Pitched-Blade Turbine (PBT) $N_P = 1.3$ $N_Q = 0.8$	Lightnin A-310 $N_P = 0.3$ $N_Q = 0.6$
Flat Two-Blade Paddle $N_P = 0.2$ $N_Q = 0.7$	Anchor $N_P = 0.6$ $N_Q = 0.5$	Retreat Curve (RCI) $N_P = 0.4$ $N_Q = 0.3$	Curved Blade Turbine (CBT) $N_P = 1.0$ $N_Q = 0.6$

Values are approximate, and will vary depending on the specific blade pitch, aspect ratio, etc.

Sources: [56, 97, 155, 169, 217, 218, 260]

influenced by impeller design style. It is usually determined empirically, based on the power input required to attain a certain Reynold's number value, and is used in a number of important calculations. If it is known, it can be used to estimate the power requirements to achieve specified mixing conditions. The various impeller types exhibit more or less characteristic values of N_P , as indicated in the table on page 2-9. Still another important group is the **pumping number**, or **flow number**, N_Q . This number, also characteristic of impeller type, gives an indication of the axial "pumping" component of the mixing action. Axial flow pitched-blade turbine impellers, for example, exhibit high values of N_Q . The figure on page 2-9 shows that N_P and N_Q are functions of N_{Re} , as is the homogeneous blend time. Since most reactors operate in the turbulent regime, N_P and N_Q can be considered constant for all intents and purposes.

Three more mixing parameters should be mentioned here. One is **energy dissipation**, E_i . This is a measure of the amount of energy transferred from the impeller to the fluid. It has units of energy per unit mass of fluid and is derived from the power number and other factors. It is very valuable for making mixing scale comparisons. Another is **specific flow** (flow per unit volume), Q/V . The third is **tip speed**, V_t , which, as its name implies, is the linear velocity of the impeller at its tip or widest point. Tip speed is particularly important in shear-sensitive applications, such as biocatalytic systems (many enzymes are shear sensitive) and in crystallizations, where it is directly related to particle attrition.

With the relationships described above, it is possible to perform theoretical calculations of N_{Re} , E_i , Q/V , etc. The best way to do this is to create a spreadsheet that performs the calculations automatically based on the values you supply. The spreadsheets can also be used to evaluate experimental data, making it easier to look at the effects of mixing speed and other changes. The major error comes from uncertainty in the values of N_P and N_Q . The typical values given on page 2-9 are only approximations, but should suffice for the first pass. The power number is the more critical of the two, and it can be determined directly for any given reactor by measuring power consumption (using a watt meter, for example) at different mixing speeds and fluid volumes. N_P can then be determined from the equations on page 2-9.

A great many other factors play a role in vessel mixing that have not yet even been mentioned. For example, the ratio of impeller diameter to tank diameter, the tank aspect (h/w) ratio, the number, shape, and location of baffles, the distance of the agitator from the bottom of the vessel, the number of agitator tiers, the effects of non-Newtonian fluid behavior, and the list goes on. It is virtually impossible to reduce all of the observed phenomena to a nice concise set of mathematical relationships. Here is a case in point. Consider the anchor-style impeller blade. Although it has a relatively low power number, (~0.6 is reported by most sources), its relative proximity to the vessel wall makes it capable of breaking up a solidified crystalline mass that couldn't be moved by many other impellers with higher power numbers. Much more information on the scale-up of mixing can be found in references [56, 188, 260].

Mixing Scale-Down Example

An aqueous-based process is to be run at a scale of 450 gal (1.7 m³), in a vessel with an 80 rpm fixed-speed CBT impeller ($N_P = 1.0$) with a diameter of 1.5 ft (0.46m). Determine how to mimic the energy dissipation E_i using a 1-L reactor with a 2" (0.05m) diameter pitched-blade turbine ($N_P = 1.3$). Assume $\rho=1000 \text{ kg/m}^3$ and $\mu=0.001 \text{ kg/m-sec}$.

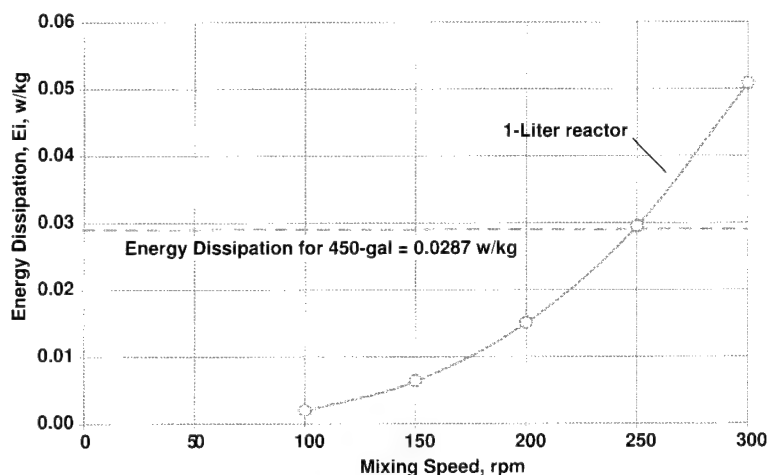
For the 450-gal reactor:

Speed (rpm)	Speed (sec ⁻¹)	N_{Re}	E_i (w/kg)
80	1.33	282133	0.0287

For the 1-L reactor:

Speed (rpm)	Speed (sec ⁻¹)	N_{Re}	E_i (w/kg)
100	1.67	4167	0.0019
150	2.50	6250	0.0064
200	3.33	8333	0.0151
250	4.67	10417	0.0294
300	5.00	12500	0.0508

Thus, the same value of E_i could be achieved in the described 1-L bench reactor by operating at approx. 250 rpm.



Charging Raw Materials

Since the hazards associated with handling chemicals are greater at large scale, always observe these basic precautions: Read the MSDS and understand the properties of the materials you are handling; wear the proper hand, face respiratory protection and protective clothing; always inert empty reactors with nitrogen before charging material of any kind.

Solvents and Solutions – Liquids can be charged directly from the drum or storage container through hoses by applying slight vacuum to the vessel. Avoid very high vacuum, which can vaporize volatile materials. Make sure condenser cooling is on and distillation valves are set to reflux. If the liquid is flammable, be sure to ground the container and bond it (connect it by wire) to the reactor or use a metal wand and hoses with metal overbraid. To avoid sucking air into the vessel as the drum empties, blanket the drum with nitrogen. More information on the safe handling of flammable solvents can be found in Chapter 6.

It is typical to mount the drum or tank on a platform balance to monitor the progress of the charge. In the interest of accuracy, it is important to weigh the sealed drum before and after charging with no hoses or ground wires attached. The feed tank can be similarly charged for later addition to the reactor.

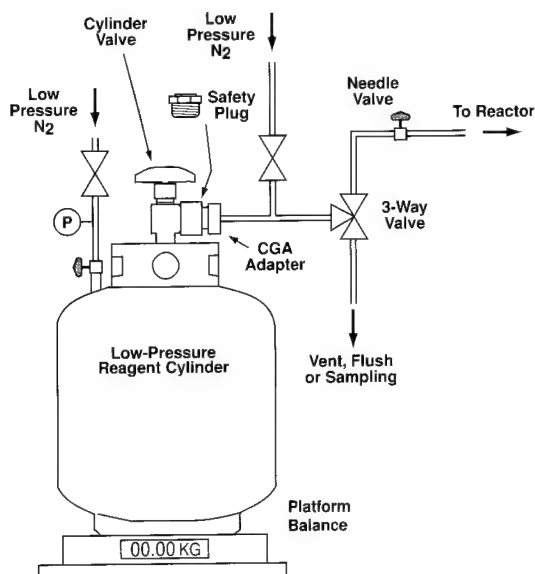
A totalizing volumetric or mass flow meter can provide a good alternative to weight measurement. It may be the only alternative when transferring directly from stationary bulk containers. Be sure that it is calibrated for the liquid in use.

For slow controlled additions use a peristaltic (tubing) pump or diaphragm pump. Peristaltics are particularly easy to use, accurate and reproducible (see page 3-11). They are probably the best choice for controlling reaction rate by metered addition of limiting reagent. When using these or other types of metering pumps, it is not likely that the reagent will accidentally be added all at once, as can happen when feeding by gravity from the head tank through a throttling valve.

Hazardous liquid reagents – Many air-sensitive, highly reactive or pyrophoric reagents are sold as solutions for ease of handling (pyrophorics are substances capable of spontaneous combustion under the right conditions, such as exposure to atmospheric oxygen or moisture). Examples include organoboranes, borohydrides, and organolithium compounds. Such reagents are usually shipped in low-pressure cylinders, and must be transferred in completely closed systems to avoid exposure to air.

Preparation is the key to safely handling hazardous compounds. Read the MSDS for the material you are using. Know ahead of time how you will deal with spills. Have an appropriate quench solution, powdered lime, or the like, handy to cover or neutralize spills. Leak test your transfer setup with inert gas before introducing the reagent. It is important to have a detailed, step-by-step written procedure available for operators to follow when making these types of transfers. Base it on the general recommendations made below, but always follow any specific instructions of the reagent supplier in handling and transferring operations.

Setup for Charging Hazardous Reagents



Use a setup such as the one shown here for discharging the cylinder contents to the reactor or for sampling. Make sure that all materials used, including valve packings, etc., are compatible. The cylinder should be securely placed in an upright position on a balance for monitoring the addition (weigh the sealed cylinder before and after the transfer to obtain the most accurate weight reading). The cylinder connection will probably be a bullet-nosed CGA-510 connection type, as used on some compressed gas cylinders (see page 7-6). Identify the exact connection type and ensure that you have the proper adapter to make a leak-tight connection with the valve manifold. Do not jury-rig this connection. Adapters should be available from the supplier. Make sure that the cylinder valve is closed tightly before removing the safety plug (note that CGA-510 and some other connection types have left-handed threads). Make sure that lines and fittings are clean

and dry before connecting. Make the connections, then use nitrogen pressure to test the setup for leaks. Make sure that the needle valve is closed before opening the cylinder valve.

Flush all lines with nitrogen to remove all air or moisture before introducing the reagent. Cylinders are usually shipped under a small positive pressure, but it will probably be necessary to pressurize it further with 10-15 psi nitrogen to discharge the contents. Use the pressure gauge to test pressure in the cylinder. Make sure that the reactor or receiver is vented or at a lower pressure than the cylinder. After the transfer is complete, blow the lines out with nitrogen, disassemble, clean and dry with care. More detailed information can be obtained from the reagent manufacturer. Aldrich, for example, publishes some excellent technical bulletins dealing with this subject [9, 10].

Gases – Gases can be charged to a reactor by subsurface sparging through a dip-tube, through a specialized sparging agitator, or by simply pressurizing the reactor head space and allowing vigorous mixing to effect the dissolution of the gas. With a dip-tube, it is important to include a one-way check valve or siphon-breaker in the addition line to prevent backup of reaction mixture into the pressure regulator or tank.

Gas charging can present a number of other challenges, for example, compensating for flowrate changes with pressure, or in the case of liquefied gases, as the cylinder cools. It is useful to know as much as possible about the solubility, thermal effects, and effect of mixing on the absorption. For more information on safe gas addition, including a typical setup diagram, and safely heating cylinders, see page 7-9.

Solids and powders – Charging solids can be a cumbersome and time-consuming operation. Usually these materials are scooped through the manway at special bag-dumping stations with filters and a blower, or charged via a closed hopper system of some kind, although even these have to be manually filled first. A few hazardous compounds, such as lithium aluminum hydride and DMAP, are available as pellets or packed in special dissolving plastic bags that can be placed directly in the reactor. This is a good alternative as long as the plastic does not introduce impurities into the batch.

The potential hazards of handling solids are often overlooked. Most organic solids form ignitable dust mixtures with air, and so it is just as important to handle them properly as it is for flammable solvents. Ignition through electrostatic discharge should be of prime concern. Even the internal friction of a falling stream of powder can generate sufficient static charge (sometimes thousands of volts) to cause sparking, spontaneous ignition and serious explosions. Nonconductive poly-drums and liners can make things worse by inhibiting dissipation of built up charges. Characteristics such as minimum ignition energy and limiting oxygen level determine the likelihood of an explosion or deflagration of dust clouds. These values are determined experimentally by a hazards analysis lab. An excellent overview of safety screening and the hazards associated with solids and powders can be found in [210].

As with any fire or explosion hazard, prevention involves removing one or more legs of the “ignition triangle” (fuel, oxygen, heat), or in this case, preventing the formation of the dust cloud, removing oxygen by inerting the system, or eliminating the source of heat or sparks.

The first step is to properly ground and bond the vessel and the powder container. This will help dissipate most of the built-up static charge, as will the use of conductive gloves and boots, or attaching a grounding strap to the operator’s wrist or his scoop. All funnels and charging chutes should be made of conductive material if possible and should be grounded. Avoid plain polyethylene bags and liners. Antistatic plastic bags or paper sacks are safer.

Also, ensure that the vessel to which the solid is being charged has been purged of all oxygen and inerted with nitrogen, argon or a similar inert gas. It follows that solids should never be manually charged into a vessel that might contain solvent vapors unless the vessel is fully inerted. For similar reasons, do not charge solids to a vessel containing flammable solvents at elevated temperatures. Seek alternative approaches early in the development program. If absolutely necessary, use an appropriate two-valve charging system or an enclosed screw mechanism and inert the vessel with a pad of nitrogen.

When charging solids manually through an open manway, it may be useful to apply a slight vacuum to the vessel or to open it to the slight negative pressure of the scrubber to prevent dust from blowing back out of the manway. However, leave a sweep or blanket of N_2 on to prevent large amounts of air from entering the vessel. If using vacuum with powders, make sure that the pump is protected with a particulates filter (see page 7-21).

Reaction Control

Chemical reactions can be characterized by kinetic rate equations that describe their temperature and concentration dependence. However, the end result of a reaction is determined not only by kinetics but also by thermodynamics. And since many reactions are reversible or involve parallel and side reactions, it is important to keep the reaction moving in the right direction and push the equilibrium forward to maximize yield and product purity. It becomes critical, then, to ensure that process operating conditions remain within the specified temperature, pressure and concentration limits. It is also important to minimize local effects that can promote side-product formation. Doing so often requires more careful planning at the pilot scale than at the bench.

Before reactions are undertaken at the pilot or kilo-lab scale, there must be a clear understanding of how the reaction end point will be determined. Does the yield peak and then begin to decline? If the reaction is prolonged to maximize yield, what is the effect on the impurity profile? The objectives must be clearly in place before starting.

Exothermic Reactions – Scaling up exothermic reactions can present significant process and safety issues. The primary challenge is to maintain constant batch temperature in spite of the heat generated by the reaction. The best way to minimize excess temperature rise is to control the reaction rate so that it is matched to the heat removal capacity of the reactor. The most common method of doing so is by controlled addition of a limiting reagent.

If the heat of reaction (per mole of limiting reagent) and the overall heat transfer coefficient (HTC) of the reactor are known, then the exact addition rate can be calculated, along with the required jacket temperature (see the example below). Heat transfer coefficient can be estimated or easily measured, but calorimetric determination of heat of reaction is vital. The alternative, of course, is to simply control the addition rate manually while keeping an eye on reactor temperature. But this is not as safe. Should this be the method of choice, however, always start the addition very slowly and be aware of the possibility that unreacted reagent can accumulate in the reactor if the reaction rate is slow, creating a potentially dangerous situation.

Even after completing the above analysis, it is common to still add a “cushion”, that is to start the addition at a lower temperature in case the temperature rises in spite of your careful estimates. However, this can create a dangerous situation by reducing reaction rate to the point where unreacted reagent accumulates in the reactor. The reagent may then react suddenly upon warming. A common guideline states that no more than 10% of the unreacted reagent should be allowed to accumulate in the reactor. A cushion can also increase operating cycle time at large scale because of the initial batch cooling time, and it may prolong addition time because of the sensible heat of the reagent.

Sometimes it is actually safer to run the reaction at a higher temperature because it reduces the possibility of reagent

Exothermic Reaction Control Example

An irreversible exothermic reaction, which generates 4700 kcal/mole reagent “Z” is to be run at 25°C (77°F) in a reactor with HTC (U) = 55 BTU/hr ft² °F and heat transfer area (A) of 16.5 ft² by controlled addition of reagent “Z”. Jacket temperature is 0°C (32°F). Calculate the maximum allowable addition rate of a 5M solution of reagent “Z”.

A first pass estimate, assuming constant U, constant heat of reaction, and ignoring sensible heat of reagent “Z” solution, can be made as follows. First calculate maximum heat removal rate (Q) using the relationship:

$$Q = \Delta TUA = (T_{\text{Reactor}} - T_{\text{Jacket}})UA$$

$$Q = (77^{\circ}\text{F} - 32^{\circ}\text{F}) \times 55 \text{ BTU/hr ft}^2 \text{ }^{\circ}\text{F} \times 16.5 \text{ ft}^2 = 40,800 \text{ BTU/hr}$$

Then, match the addition rate (\dot{V}) of Reagent “Z” to this heat removal rate:

$$\dot{V} = \frac{Q}{\Delta H \times [A]} = \frac{40,800 \text{ BTU/hr}}{18,600 \text{ BTU/mol} \times 5 \text{ mol/Liter}} = 0.44 \text{ L/hr} = 7.3 \text{ ml/min}$$

accumulation. Operating the reaction at reflux, for example, may be advantageous because reflux is usually capable of removing heat from a system at a rate many times higher than the reactor jacket, and thus offers excellent supplemental cooling. On the other hand, operating at reflux is energy intensive and limits the maximum reaction temperature to the boiling point of the solvent.

The controlled reagent addition is best accomplished using a peristaltic or other metering pump. A pressurized tank on a platform balance, with flow controlled by a needle valve, is also used quite often. Feeding by gravity from a head tank is not recommended because of the possibility of valve failure or error leading to accidental addition of the reagent all at once. If selectivity or purity is affected by rapid reaction at the point of addition, it may be necessary to add the reagent subsurface, spray it into the reactor, or prepare a more dilute solution for addition. As an additional safety measure, have condenser coils cooled and distillate valves set to reflux before beginning addition. Then, if there is an adverse event, maximum cooling is already available. Also, of course, good agitation is required to ensure that the reagent is able to react as it is added.

Whenever possible, prepare solid reagents by dissolving them first, since liquid addition is much more easily controlled. As a general rule, controlled addition of solids is extremely difficult at scale.

Catalytic Hydrogenation – This is a very common type of reaction but one that poses unique safety risks. Typically, the reaction is run at elevated temperature and pressure under an atmosphere of hydrogen in the presence of a noble metal or similar solid catalyst. The most obvious safety issue is the use of hydrogen gas, which is odorless, colorless and extremely flammable over a wide concentration range. Handling the potentially pyrophoric catalysts can also present safety issues, which can be minimized by using water-wet grade catalysts. It has already been said that adding the catalyst to a reaction mixture last is not acceptable. Catalytic hydrogenation is usually exothermic, and adding catalyst last allows no possibility of control. The best method is to use the hydrogen feed rate as the control system. If temperature begins to climb out of range, shutting off the hydrogen supply usually brings the reaction to a stop. Alternatively, the reaction can be controlled by slow addition of another limiting reagent.

The variables that can be investigated to optimize hydrogenation are catalyst amount, operating pressure and temperature, and mixing rate. Generally, reaction rate is more sensitive to changes in temperature than pressure, but both can be used to adjust rate or selectivity. Because this is a heterogeneous reaction, mixing plays a critical role. In fact, if hydrogen is simply introduced into the reactor head-space, it becomes a three-phase mixture and mass transfer becomes extremely critical. In general, the highest possible degree of mixing should be used. Hydrogen should never be introduced into the system without agitation. Commercial hydrogenation vessels are specifically designed to maximize gas/liquid mixing.

Dehydration Reactions – Certain reactions, such as imine condensations, result in the elimination of one or more moles of water. They are thus called dehydrations. Usually, when the water reaches its concentration equilibrium, the reaction will stop. To drive the reaction forward, water must be driven off or otherwise removed from the system. This is most often accomplished by azeotropic drying. Both homogeneous and heterogeneous azeotropes can be very effective in removing water. In the case of heterogeneous azeotropes, a *Dean-Stark* apparatus may be used in the reflux return line. This is a water-trapping device that can be relatively easily scaled up as long as accommodations are made in the reactor train ahead of time. It should be taken into consideration when reaction equipment is designed or specified. In many cases, the amount of water collected can be used to monitor reaction progress. More information on azeotropes and a list of solvents that can be used for drying can be found in Chapter 6. Water can also be removed from the system by circulating the mixture through a fixed bed of solid dehydrating agents such as molecular sieves. However, this is more cumbersome and expensive and so, if at all possible, the reaction should be designed to use azeotropic drying.

In-Line Reaction Monitoring and Control – It is often possible to follow the progress of a reaction by directly monitoring some parameter by means of in-line instrumentation. Some common examples include pH, conductivity, or infrared absorbance. This last method can be an extremely powerful technique for process development where applicable. Systems such as the React-IR® manufactured by ASI, once properly calibrated, enable the user to observe the formation and disappearance of various chemical species in real time.

For a brief survey of other common reaction types encountered in chemical processing, see page 8-20.

Sampling for Reaction Monitoring

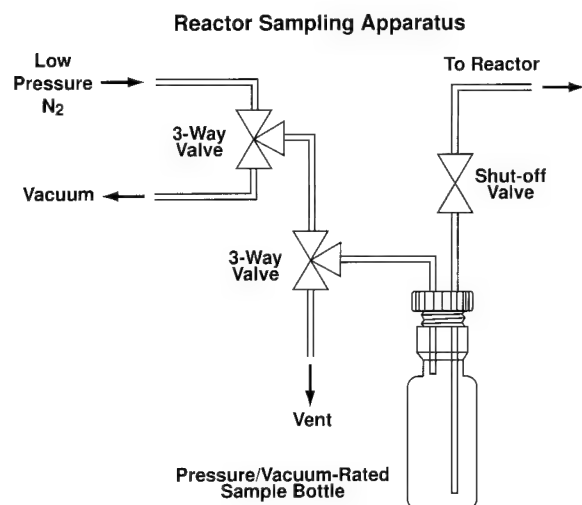
For most chemical reactions, it is necessary to set some criteria to determine if the reaction is complete or proceeding as expected. This should never be based on elapsed time. It is important to have an independent measurement based on physical parameters, and thus it is typical to collect samples for analysis at selected points in the process. Additional samples may be collected and stored for later use in waste stream analysis, mass balance closure, and stability studies.

Improper sampling technique is one of the greatest sources of error during in-process checks, especially for non-homogeneous mixtures and slurries. Differences in conditions between the reactor and the sample bottle can introduce significant variability. Therefore, sampling must be well thought out. A sampling plan should be in place before starting any batch. Understand when the samples are to be collected, how they are to be treated, quenched, stored, frozen, etc. Ensure that analytical support is available if processing decisions must be made based on results. It should also go without saying that each sample must be assigned a unique identification number and logged into the batch record, or better, the sample log book (see page 1-5). Failure to take this simple step can cause confusion and loss of important data later on.

Sampling Apparatus – Collecting representative samples from the reactor is critical, and this must usually be accomplished without opening the reactor to limit risk to personnel and to avoid introducing air into the batch. A sampling set up like the one shown below is highly recommended. This convenient apparatus enables safe sampling of reaction mixtures while the reactor is agitated at elevated pressure and temperature and makes it easier to backflush the sampling line with solvent between samples. By using a large enough sampling line and shut-off valve, slurries and emulsions can also be sampled easily. For sampling two phase mixtures, it is usually easier to sample both phases with vigorous agitation and allow the phases to separate in the sample bottle.

Use bottles that are pressure or vacuum rated as necessary, as well as PTFE tubing where needed to better protect against corrosion and contamination of the sample. Ace Glass, Inc. manufactures hydrogenation bottles that are ideal for this purpose. Other materials should be PTFE or stainless steel. Note that the sample may also be hot! Be certain that the vent is aimed away from personnel. Support the entire apparatus well so that it doesn't take three hands to operate it. The best idea is to construct a permanent apparatus and mount it securely on or near the reactor.

It is not necessary to use three-way valves, but doing so simplifies using the device. The sample is drawn into the bottle by applying a small amount of nitrogen pressure on the reactor or applying a slight vacuum on the sample bottle. To blow excess sample back into the reactor or to clean out the line, apply nitrogen pressure on the sample bottle. Have a sample bottle full of clean solvent handy for flushing the line back to the reactor. Note that this type of device also works well for introducing seed crystal slurries and similar materials.



Other Sampling Methods – It is possible to collect samples through the reactor bottom valve, but this is not recommended because it is not easy to control the amount collected and a considerable amount of material may need to be flushed through the valve to ensure that the sample is truly representative. Also, undissolved solids tend to settle to the bottom valve.

Samples may also be collected directly through the manway using a dipper or a grab device that accepts threaded bottles. Avoid using glass bottles for this purpose. Some grab devices allow the bottle to be opened from the operator end, allow it to be dipped into the liquid phase of choice, opened for sampling, and then sealed and withdrawn. But the dangers of inserting anything into a reactor while the agitator is operating are obvious and opening the manway in general can compromise the integrity of the batch and operator safety. Sampling via the manway is sometimes simply not an option in some situations, such as pharmaceutical processing or other GMP environments.

Workup

Workup is a general term encompassing the downstream processing steps that follow completion of a chemical reaction. The ideal workup is one in which the reaction mixture is cooled, crystallized and filtered. But in the real world, this is often not the case. A more typical workup includes addition of a second phase to quench the reaction, and extraction steps to remove unreacted starting materials or unwanted impurities, before concentrating and isolating the product or carrying the stream on to the next step. This is a point where many things can, and often do, go wrong.

Reaction Quench – Quenching neutralizes the reactive species to stop the reaction and can minimize the formation of impurities. Quench solutions are usually aqueous, which can be convenient for subsequent phase separation, but caution must be used when adding water to a mixture that contains water-reactive compounds. The quench must be added slowly with good agitation to keep the layers well mixed and dispersed for good interfacial contact, and to prevent the accumulation of unquenched species. Cooling may need to be applied to the reactor. Precooling the quench solution or the batch can also help minimize temperature excursions. In some cases, for example when quenching lithium reagents, it is common to quench with acetone first followed by aqueous washes.

Any number of quenching agents are commonly used, including citric acid, hypochlorite, bisulfate, bisulfite, hydroxides, carbonates, mineral acids, ammonia, various buffers and so on. Take care to ensure that salts formed during the quench will not precipitate and interfere with the phase separation. Optimize the concentration of the quench solution to minimize waste. A good survey of quenching agents for various species is given in [11].

Often, reactions at the bench are quenched by pouring them *into* the quench solution. This avoids generating excessively high concentrations of any solutes that have a high affinity for water during the beginning stages of aqueous addition, which can sometimes affect yield or the impurity profile. But using this approach at the pilot scale is more problematic and requires additional transfers or the use of a second vessel. Adding the quench to the batch is preferred.

Extraction and Phase Separation – Extraction is a very common operation usually carried out to remove impurities from the product stream. A simple aqueous extraction that works well at the bench can turn into an emulsion nightmare at larger scale because of differences in agitation and the greater geometric distances that materials have to migrate as the dispersion breaks. Interestingly, large-scale batch extractions are often characterized by the presence of an ugly “rag layer” at the interface, consisting of dust, carbon black, and other insolubles that may have been in the raw materials or were left behind from previous operations, that is almost never observed at the bench. It should be clearly understood before starting the operation whether the interfacial material is to be discarded with the extract phase or retained.

The extraction operation must be well-designed from the beginning if it is to scale well. It is useful to know the partition coefficient of the substance being extracted, the speed of the extraction and its dependence on mixing, as well as the densities and polarities of the two phases. Among the most difficult reactions to work up are those containing water reactive components, such as strong acids, chlorinating agents, etc. In these cases, great care must be taken during the addition of any aqueous quenches to avoid dangerous exotherms. It may be possible to use a smaller stoichiometric amount of the troublesome agent from the beginning, reducing the amount of excess to be dealt with during workup.

pH can be important for good extraction and separation, and it should be determined if pH needs to be measured and adjusted in the pilot reactor. It is also common to extract a mixture with acid to remove alkaline by-products, and then with base to remove acidic by-products, with a water wash in between. This is colloquially referred to as an “acid-base flip-flop”. Sometimes an immiscible organic solvent makes an effective extract phase.

Most of the factors that can help simplify extractions in pilot equipment are a matter of common sense. Turbulent mixing is critical for generating high surface area for extraction, but if agitation is too high, the batch can emulsify. Better to mix longer at a lower speed than to risk an emulsion. Keeping the batch within temperature limits is often overlooked. Many times, batch temperature will drop because the added water is cold, or sometimes because of a large positive enthalpy of solution. Be sure to allow sufficient time for the phases to fully separate. Installing a “lantern”, a double-valved sight glass, at the bottom of the reactor can help ensure clean cuts at the interface. Place a flashlight behind it to improve visibility. Once the bottom phase has been drained off, it is good idea to “bump” the agitator – to turn it on very briefly then off again, to sweep remaining water droplets down to the drain valve. Then let it settle and drain again.

Partition Coefficient and Extraction Efficiency

Amount of Total Solute Remaining in Raffinate Phase			
	$K = 5$	$K = 10$	$K = 20$
1 extraction ($V=1$)	16.7%	9.1%	4.8%
2 extractions ($V=1/2$)	8.2%	2.8%	0.8%
3 extractions ($V=1/3$)	5.2%	1.2%	0.2%

$$\text{Fraction of total solute remaining in raffinate after any one extraction} = \frac{1}{1 + K \left(\frac{\text{Wt. extract phase}}{\text{Wt. raffinate phase}} \right)}$$

Certain solvents exhibit properties that make them problematic for aqueous extraction. The most notable is CH_2Cl_2 . The specific gravity of CH_2Cl_2 is very close to that of water, and in some situations, due to slight changes in temperature or concentration, the phases in an extraction can switch position, leaving the aqueous waste stream on the top, and the product solution on the bottom. This is one reason why it is advisable to collect extract phases in clean drums, so that the stream can be recovered if an error is made.

Partition Coefficient – Consider a typical case where an organic solution is being extracted with water. Hopefully, most of the material of interest migrates into the aqueous phase but a finite amount remains in the organic phase. In extraction terminology, the aqueous is called *extract phase* and the organic, the *raffinate phase*. The efficiency of the extraction can be expressed by the partition coefficient, K , the ratio of the concentration (in weight percent) in the extract to that in the raffinate:

$$K = \frac{\text{wt\% in aqueous (extract phase)}}{\text{wt\% in organic (raffinate phase)}}$$

This ratio remains constant regardless of the extract volume, and thus a simple mass balance will show that for a given total weight of extract, many small extractions are more effective than one big extraction. This is demonstrated in the table at the top of the page. In each of the examples given, the total weight of the extract phase is equal to the weight of the raffinate phase. The table shows that by using the same total extract volume, but splitting it into two or three smaller extractions, the overall efficiency is greater, with less and less of the solute remaining in the raffinate. This, of course, has to be weighed against the time, effort and expense of additional extraction steps. Also shown is the equation for determining the fraction of the starting amount of solute left in the raffinate after one extraction step, as a function of partition coefficient.

Problem Separations – As mentioned above, it is not unusual for pilot extractions to turn into emulsions that will not separate even after hours of waiting. Some of the more common “tricks” for separating emulsions are mentioned here. Applying heat or cycling the temperature up and down can help break the emulsion. Sometimes very gentle movement of the agitator helps. As the phases begin to separate, it may help to drain off the bottom layer in portions as it accumulates, but patience can be a real virtue in these situations.

Adding a small amount of a co-solvent, such as methanol, can break some emulsions. Adding salt will increase the density of the aqueous layer and therefore the difference in density between the two layers. Avoid adding too much, or the bottom valve could become clogged. Should this happen, apply steam or some other *safe* source of heat to the valve to dissolve the blockage. In other cases, alternately applying vacuum or slight pressure can speed separation.

Very often the emulsion is stabilized by the presence of fine particulates. These can be removed by polish filtering the entire batch through a cartridge filter of say 0.5μ (0.5 micron) pore size and rinsing out the vessel before recharging the batch. pH adjustment may also break an emulsion, particularly when aqueous bases are used with toluene or CH_2Cl_2 .

Occasionally, the interface is so clean that it is impossible to see. In such cases, a conductivity measurement can be most useful to distinguish between the aqueous and the organic phase. It is also useful to sprinkle a pinch of Celite (diatomaceous earth) into the batch. The celite tends to collect at the interface and helps to accentuate it. The celite of course must be removed later by polish filtration, and so this is not recommended during the final processing steps.

Polish Filtration / Decolorization / Water Removal

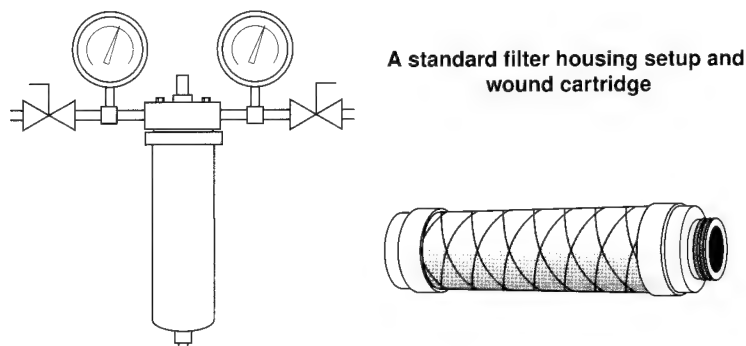
Prior to crystallization and isolation of a product, and in other instances as well, it is often necessary to pass the solution through a fine-pored filter to remove residual undissolved reagents, dust, celite or other particulates that may have been introduced in the raw materials. This process is called polish filtration or sometimes clarification. The standard approach is to pump the solution through a cartridge filter. Filter cartridges come in many standard shapes, sizes and capacities, with different surface areas and pore sizes. Major manufacturers include Cuno, Filterite, and Pall.

Cartridge choice depends on the nature of the batch and how much solid material is present, but as a general rule, a typical 100-L batch can usually be easily filtered through a standard 10-inch cartridge. Other standard sizes are 4", 20" and 30". Manufacturers suggest that cartridge filtration be used for batches that contain less than 0.1% solids. Normally, a pore size of 5 μ is adequate for general chemical processing, but cartridges are available with pore sizes down to 0.1 μ . Filter cartridges are also made of many different materials, such as cotton (cellulose), polypropylene, polyester, and nylon. The compatibility of cartridge materials, cartridge O-rings, and housing seal gaskets is critical and must be determined before use.

It is usually simplest to keep a freestanding filter housing and flexible lines available for connecting to process vessels. In other cases, the filter may be permanently piped into a system, in which case service valves and a bypass line are necessary. No matter what the setup, include upstream and downstream pressure gauges to monitor batch progress and to help spot a plugged filter. A maximum allowable pressure differential across the cartridge of about 30 psig is typical, but check with the manufacturer if in doubt. Pressure drop will usually increase as the filtration progresses and the filter catches more material and begins to plug. Pressure drop and flow rate also vary depending on pore size, surface area, and the nature of the filter medium (pleated vs. wound, for example). Wound cartridges, often called "depth filters" usually have more capacity.

Decolorization/Drying – It is common laboratory practice to reduce the amount of color in product solutions by stirring in some powdered carbon black (activated charcoal) or to dry solutions by mixing in solid drying agents, such as MgSO_4 or others (see page 8-18 for a list of common drying agents). These materials are insoluble in the solutions for which they are used, and they need to be removed by filtration. Special bag filters or polishing filters, such as those manufactured by Sparkler, Inc., are available for this purpose at large scale. However, these units can be expensive. Also, it is not desirable to add these solid agents, especially carbon black, directly to a multipurpose chemical reactor because of the difficulty in removing them later. Cleaning a reactor of all traces of powdered carbon can be a nightmare, because it is not soluble in anything. Soap and water often proves to be the best cleaning solution. Some facilities designate a simple mixing tank just for decolorization operations. The exception of course is in cases where precious metal catalysts on carbon substrates (Pd on carbon, for example) must be used in a reaction, in which case adding it to the reactor is not a matter of choice. Removal of powdered carbon by filtration requires a pore size of at least 0.1 or 0.2 μ . Even then, to retain submicron carbon dust particles, it is often necessary to pre-coat the filter with a filter aid such as Celite (diatomaceous earth), Perlite (silica), or Solkaflor (cellulose), for better retention. A cake of diatomaceous earth has a very open pore structure, allowing for fast filtration, but the tortuous nature of the paths through the cake help trap small particles. Filter aids come in a variety of pore sizes for different applications.

The disadvantages of using filter aids is that they are often inconvenient to work with, and could possibly contaminate the batch. A good alternative for the pilot scale is to use filter cartridges that contain the filter aid, decolorizing agent or drying agent within them. The batch can be passed or circulated through the filter to achieve the desired effect without having to dirty the vessel. This approach should be tested at the bench if possible before applying it at the pilot scale since it may not be as effective as mixing and filtering in all cases. Many manufacturers make small-scale filter capsules that are useful for testing at the bench.



Batch Distillation

Distillation covers a number of operations involving boiling and vaporizing a liquid, and then separating and condensing the vapors. One way to carry out a distillation is to simply remove the condensing vapor so that none of it returns to the boiling pot. The other way is to ensure that a portion of the condensate is directed back to the boiling pot in such a way that it is intimately contacted with the vapor rising from the pot, which is called operating with **reflux**. Reflux is used in many multistage distillation columns for separating multicomponent mixtures. The term reflux is also sometimes loosely used to mean “full reflux” by which distillate is simply returned to the pot continuously to allow for extended boiling of the mixture without loss of solvent.

We will focus here on single stage batch distillation without reflux, which is sometimes called **flash distillation**. This is the type of operation most commonly encountered in batch pilot reactors for volume reduction, solvent exchange, or removal of residual water by azeotropic drying.

Vacuum vs. Atmospheric Pressure – One of the key decisions to be made early in process development is whether a distillation operation will be carried out at atmospheric pressure or under reduced pressure (vacuum distillation). One of the most common sights in a chemical laboratory is a chemist reducing the volume of his reaction on a roto-vap (rotary evaporator). It is quick, convenient and simple to operate. And while vacuum distillation is an important and widely used technique, it is somewhat more costly and difficult to control, and thus not employed at the manufacturing scale unless necessary to prevent the degradation of heat-sensitive compounds or the formation of impurities, or because the boiling point of the solvent is so high that it cannot be removed otherwise. The table below lists the major advantages and disadvantages of vacuum distillation vs. distilling at atmospheric pressure. It should be mentioned that there are pilot scale (up to 100-L) roto-vaps that enable a process to be scaled up quickly when necessary. They are a useful addition to a pilot plant, but the decision about distillation conditions for long-term scale-up needs to be a deliberate one.

Solvent Exchange – One of the more common uses of batch distillation is the exchange of one solvent for another. This is necessary when one solvent is better suited for, say, running a reaction and another is better suited for crystallization. When there is a large difference in boiling point or a favorable azeotrope exists, it is easy to add the second solvent and then distill until the first solvent is all removed. Some cases are more involved and may require using a third solvent as an intermediate or as a component in a favorable ternary azeotrope (an entrainer). To make this determination, it is necessary to have information on the vapor pressures of the solvents involved as a function of temperature, and the nature of their vapor-liquid equilibrium relationship (see page 6-25). Excellent references on this subject are [61, 234].

Operating Tips – Further general information about distillation, phase equilibria, the nature of azeotropes, and a list of common azeotropes can be found in Chapter 6. The remainder of this section will be devoted to offering recommendations and pointers for more effective operation of batch distillations.

Many pilot reactor have glass condensers. Know the pressure limits of the condenser and throttle the operating pressure of your coolant loop accordingly. Although it is rare for glass condensers to burst under pressure (design specs always

Advantages and Disadvantages of Vacuum Distillation

Advantages	Disadvantages
Lower temperature distillation, which is safer for heat-sensitive compounds.	Additional capital and operating cost of vacuum pump and control system.
Possibly higher solvent removal rates because of greater ΔT between the jacket and the pot. This is especially important where utility issues limit jacket temperature.	Possible introduction of air leaks and subsequent explosion hazard when using flammable solvents.
Allows operation when not otherwise possible if jacket temperature is limited.	Requires colder condensing temperatures or greater condensing surface area.
Possible energy savings and reduced heat loss to surroundings while operating at lower jacket temperature.	Inverse relationship between ΔH_{vap} and temperature requires greater energy input at lower temperature.
Enables removal of extremely high-boiling solvents.	Greater foaming problems.
	Lower vapor density means lower mass transfer rate and theoretically longer distillation times.

include built-in safety margins), this is an extremely important consideration if you are using water-based coolant and water-reactive reagents in the reactor.

Pay attention to the melting point of the solvent you are distilling and the condenser operating temperature. Cyclohexane, for example, freezes at 6°C, well above the temperature at which condensers are usually operated for other solvents. The frozen solvent could easily plug condensers or small lines. Also, be aware of the need for secondary condensation, such as a dry-ice/alcohol trap, when vacuum distilling low-boiling solvents, since there will probably be considerable loss of vapor through the primary condenser. This can also lead to emission of flammable vapors at the vacuum pump exhaust. A vapor knockout trap should be installed at the pump outlet as standard practice (see page 7-22).

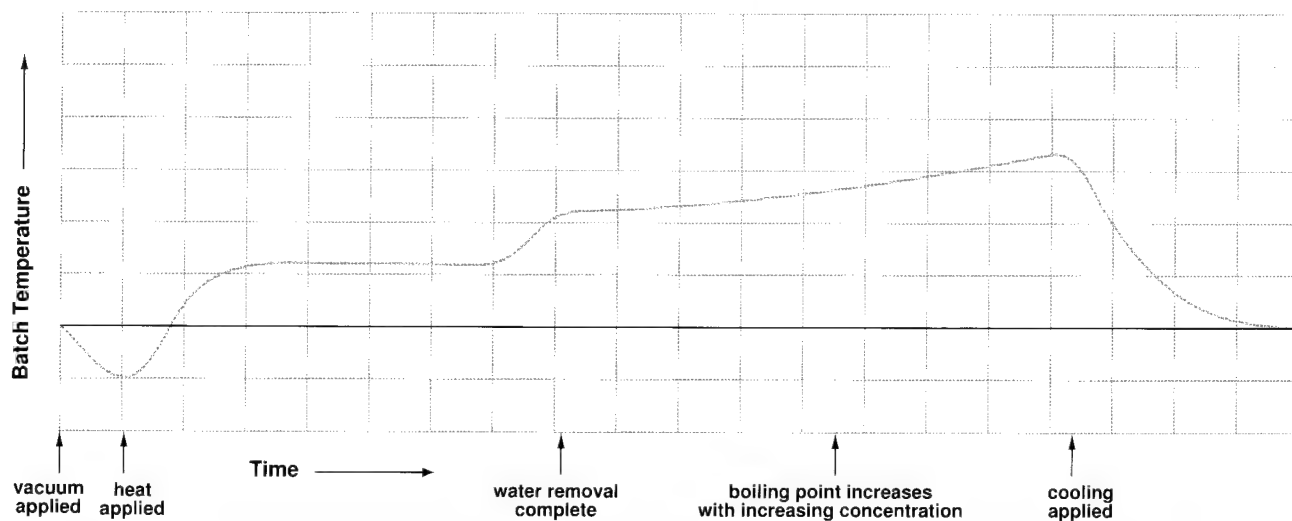
Know the approximate boiling point of the solvent or solvent mixture you are using at your intended operating pressure. This will help to fine-tune the conditions more quickly and help avoid surprises. If you are using vacuum, the charts on pages 6-22, 23 can help estimate the boiling point at reduced pressure, although other components in the mixture will have an effect. Check for the existence of a known low-boiling azeotrope (page 6-28, 6-40). If the purpose of the distillation is azeotropic removal of water, or removal of one solvent from a binary mixture, a sudden increase in boiling point can indicate that the water or solvent is completely removed (see the chart below), but to be certain, samples should be collected and tested. A sample apparatus such as the one shown on page 2-15 can be used even during vacuum distillation without interrupting the process. A device can also be set up to sample the distillate stream before it enters the receiver. Then gas chromatography (GC) or even refractive index, a simple but highly sensitive technique, can be used to determine the distillate composition.

Have a checklist tailored to your particular setup to ensure that before you start the distillation, all valves are in the correct position, that the heating, cooling, inerting and vacuum utilities are operational and that no maintenance is scheduled for them. Ensure that coolant is flowing at the proper flowrate and temperature before applying heat or vacuum to the reactor. Make sure that there is sufficient room in the receiver to collect the distillate.

Agitation should be set as high as practical to maximize heat transfer. Apply heat slowly to prevent flooding the condenser by exceeding its capacity. If using vacuum, try to establish the desired pressure in the reactor first (a vacuum controller is a must), and then *slowly* apply heat to the reactor. This will help minimize foaming. A very small nitrogen bleed into the reactor can also be helpful to knock down a high head of foam. When stopping the distillation, always shut off the heat first. Then, once the batch is cooled, vacuum and agitation can be turned off.

Monitor the temperature in the reactor during distillation, ideally with a strip-chart recorder or digital computer interface that offers a visual display. This makes it easier to detect changes in conditions and monitor the progress of the batch. It's also a good record to have for troubleshooting later on. The figure below shows a typical temperature profile for a batch distillation.

Temperature / Timeline of a Typical Batch Distillation



Crystallization

It has been said that in crystallization, the “solvent is everything”. Solvent *is* important, but the effects of mixing, cooling profile and co-solvent addition rate can also have a significant impact on physical properties such as crystal size distribution, morphology, and polymorph. This makes crystallization one of the most difficult operations to scale up. At the same time, generating a product with consistent particle size, crystal habit, polymorph or solvate is becoming an increasingly important aspect of quality control. **Crystal habit** refers to the macroscopic physical shape of the crystal – for example, needles, cubes, or plates. **Polymorphism** is the tendency of some compounds to exist in more than one stable crystal lattice arrangement. **Solvation** refers to the stoichiometric inclusion of solvent molecules into the crystal lattice (called *hydration* when the solvent is water).

A great many organic compounds can exist in more than one crystal form. Sometimes the presence of trace impurities can give rise to different forms. A solid product or intermediate that exhibits polymorphism or that has different solvated forms can be problematic since the different forms will likely possess different properties. Melting point, stability, reactivity, solubility, rate of dissolution, and bioavailability can all be affected, as well as bulk density and powder flow characteristics. Consistently producing a given form at the bench is often challenging enough, but maintaining that consistency as the process is scaled up is difficult because many controlling factors cannot simply be scaled up linearly. There are many crystallization processes that actually behave better at large scale than at the bench, but getting to the large scale requires understanding the critical factors affecting the process. It helps if the crystallization was designed at the bench with scale-up in mind. A well-designed crystallization can improve product purity and uniformity, eliminate additional purification steps, and reduce cycle time by providing faster filtration and better drying behavior. Because of the impact of particle behavior on subsequent processing steps, crystallization is best viewed not as an isolated step, but as part of an integrated series of unit operations.

Bench Studies – Identifying the scale-up parameters that control crystal habit can be a lengthy undertaking, but the more that is understood about the process at bench scale the better the chances of successfully scaling up while still maintaining the desired particle properties. Studies carried out in automated bench reactors with microprocessor-based temperature control can be invaluable in understanding the influences of supersaturation, nucleation, cooling rate and mixing effects. These units usually use turbidity or laser particle detectors to monitor the progress of the crystallization while automatically controlling temperature, concentration, agitation speed, pH or other parameters selected by the experimenter. A great deal of information can be obtained in a relatively short time. See [212] for more information.

One of the most important pieces of information is the solubility of the product vs. temperature. The solubility curve should be established over a reasonably broad temperature range before undertaking any crystallization work. Here again, automated reactors can complete a solubility curve in less than a day with no attendance. When measuring the solubility manually, be sure to look at no less than 3 temperature points, and allow sufficient time for the mixture to establish equilibrium before sampling. It is common to plot the solubility on a semilog plot as wt% vs. $1/T$. For most systems, this should generate a straight line, making extrapolation to other temperatures easier. See the example on the following page.

Early work should be directed towards developing a crystallization process that is well-controlled and proceeds “smoothly”. Crystallizations that crash out of solution rapidly due to high degrees of supersaturation will not be scalable. It’s easy to crank up mixing speed in a round-bottom flask to break up a logjam, but another thing altogether to achieve those power/volume input levels in pilot and manufacturing equipment. Also, other things being equal, the easiest crystallizations to scale up are those involving only a single solvent. Then only temperature, concentration and mixing need be manipulated. A further advantage is that it is generally easier to strip and recover a single solvent than a mixture of solvents. Don’t get bogged down worrying about solvent recovery too early in the development effort, but in any event, the fewer solvents used the better, for many operational and quality control reasons.

As a starting point, look for a system that exhibits a solubility of about 10wt% at the high temperature or at starting conditions and about 1wt% at the low temperature or at end conditions. By mass balance, the yield in such a case should be roughly 90%. See the example for estimating yield based on solubility on the following page. Note that the solubility data must be put in terms of g/g solvent for correct yield calculations. Another parameter commonly referred to in the industry is the **slurry density**. This is the amount of solids per unit mass of slurry. Many commercial crystallizations

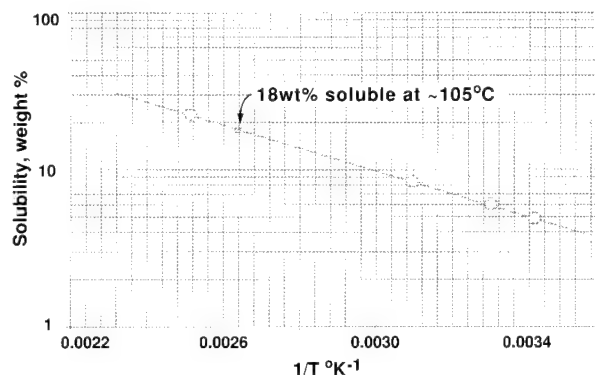
Crystallization Yield Example

Solubility data for compound "X" is given in weight % vs. temperature in °C. Use the data to estimate the crystallization yield of "X" if starting with an 18 wt% solution at saturation and cooling to 20°C. Determine the slurry density at the isolation temperature.

Raw Data		°K	1/°K	solubility g/g solvent
°C	wt%			
125	22.5	398	0.00251	0.290
50	11.5	323	0.00310	0.130
30	5.9	303	0.00330	0.063
20	2.0	293	0.00341	0.020

Plot wt% vs 1/°K as shown to estimate that the 18 wt% solution must be heated to ~105°C to fully dissolve.

The data must then be converted to solubility in terms of g/g solvent to determine the yield and slurry density as shown below (18 wt% = 0.22 g/g solvent):



$$\text{Yield} = \frac{\text{starting concentration (g/g solv)} - \text{final solubility (g/g solv)}}{\text{starting concentration (g/g solv)}} = \frac{0.22 - 0.02}{0.22} = 0.91 \text{ (i.e. 91\% yield)}$$

$$\text{Slurry density} = \frac{\text{starting concentration (g/g solv)} - \text{final solubility (g/g solv)}}{\text{starting concentration (g/g solv)} + 1} = \frac{0.22 - 0.02}{0.22 + 1} = 0.164 \text{ (i.e. 16.4\%)}$$

operate at slurry densities up to 30% or higher. Such high densities, when possible, result in more efficient filtration and vessel utilization and less waste in the form of mother liquors and washes. Depending on the nature and amount of impurities, the filtrate and wash may be saved, pooled and concentrated for a second-crop.

Make sure that the crystal slurry is stable. Avoid "kinetic crystallizations", such as the preferential crystallization of an enantiomer that must be harvested before reaching equilibrium. These will never scale reliably, since operating times are so greatly expanded in pilot and manufacturing plants. It can easily take more than 24 hours to isolate a batch at scale.

Supersaturation – The degree of supersaturation is the driving force for crystallization. It can be controlled by cooling rate, addition of anti-solvent, addition of acid or base, or other means. High supersaturation means faster crystallization and a higher chance for occlusion of solvent or impurities in the crystal lattice. Strive to keep the driving force constant, at no more than 10-20% of the solubility at any point. Fine temperature control is needed to achieve this.

In reactive crystallizations (or precipitations) the degree of supersaturation is a function of reaction rate. In this case, the solid product is usually quite insoluble in the reaction medium. Better control can be obtained by lowering the concentration or temperature to reduce the reaction rate. Sometimes, crystallization is driven by the thermodynamics of the chemical system, and continuous removal of crystalline product from solution can help drive the equilibrium forward.

When salting out aqueous solutes by acid/base titration, monitor the pH to better identify the precipitation endpoint. Determine the pKa of the product. It may be possible to optimize a precipitation step or improve purity by controlling the pH. Vigorous agitation is extremely important in this application. For more on pH control see page 5-23.

Cooling profile – Cooling is the most fundamental way to control supersaturation and thus crystallization rate. The cooling rate must be well matched to the crystal growth kinetics. Fast cooling in a case where crystal growth is slow results in a high degree of supersaturation and possibly crash crystallization. The figure on page 4-4 shows three possible batch cooling profiles. Many crystallizations are successfully scaled up using the "controlled nonlinear" profile shown in the top curve, which is designed to maintain a constant degree of supersaturation throughout the crystallization. Early on, cooling is slow to allow a good seed bed to become established. Later, as more and more material comes out of solution, cooling rate can be increased. This is the same general shape of the curve for addition of anti-solvent when that is the method used to control supersaturation. The exact curve shape is determined by supersaturation studies. When programmable control is not available, the curve can be approximated by a series of linear temperature changes. As a

general rule, slower cooling results in larger particle size, faster cooling in smaller particle size. Due to the logarithmic nature of the solubility curve, changing the starting concentration can change the end result of a given cooling profile. Some batch crystallizations can be carried out at constant temperature, once crystal growth is established, but more often, the optimum temperature for crystal growth is not the same as that for seeding and nucleation. Monitoring the batch temperature with a strip chart recorder or similar visual display is very useful. Crystallizations and precipitations may be exothermic or endothermic and crystallization onset may be detected by small temperature changes.

Nucleation and seeding – Nucleation is a critical event in the crystallization cycle. Many solutions will nucleate spontaneously at supersaturation or upon aging, but this leaves no possibility of controlling the nature of the crystals obtained. A better way is to intentionally seed the reactor with seed crystals that have the desired properties, either by introducing dry seeds or a seed slurry to the solution, just at the point of supersaturation. Seeds must be very well characterized, and seeding can mean one more raw material to deal with, but the value of the technique can outweigh these disadvantages. Seeding can sometimes help establish the particular polymorph that will be obtained. However, it is usually not the sole determinant of the final particle size distribution. Particle size is strongly dependent on crystal growth rate, which is driven by the degree of supersaturation, and thus by cooling or anti-solvent addition rate and other factors such as attrition (crystal breakage) and agglomeration.

It is important to ensure that all solids are dissolved prior to seeding. The amount of seed to use can vary from 0.1% to more than 2% of the total product. Preferably, the product from standard batches can be used as seeds. The disadvantages of having to operate special batches for the production of seeds are obvious. Some experts feel that the best source of seeds is a sample of the crystal slurry from a previous batch, because it consists of material covering the full particle size range. Others recommend the use of fines rejected from sieving operations [271].

Mixing Effects – Good mixing is absolutely necessary to eliminate the possibility of temperature gradients and “hot spots”, but beyond that, mixing parameters such as Reynold’s number, agitator tip speed (shear) or power input (energy dissipation) can all play important roles. Good agitation is necessary to keep solids in suspension and prevent agglomeration. Even with adequate mixing, cold wall temperatures can result in the formation of a rind on the inner wall that can drastically reduce heat transfer rates. This rind can sometimes be melted off by applying a quick burst of heat to the jacket (sometimes called “flash heating”). When adding anti-solvent, good mixing can help minimize concentration gradients that can cause crash crystallization and the build-up of a solid crystal mass on the agitator, baffle or solvent addition tube. Certain impeller styles, such as a pitched-blade turbine, are particularly well suited for keeping solid slurries in suspension. Such an agitator is recommended for vessels that will be designated for crystallization operations, but it is not necessarily the best impeller for all other operations. Adding a small second impeller near the bottom of the vessel can also help keep thick slurries well mixed, as can the addition of more baffles. Be aware that high-speed mixing or high-shear impellers can significantly increase attrition, especially when the product slurry must be held during lengthy isolation operations. For more on mixing effects in crystallization see [103].

Evaporative Crystallization – This is a useful technique that can overcome the minimum mixing level limitation often encountered when a large volume must be concentrated down for crystallization. The solution to be concentrated is continuously fed from the original vessel to a smaller crystallizer vessel, while solvent is stripped from the crystallizer by distillation so as to maintain a constant level. The disadvantage of this approach is that it requires the use of a second vessel. However, it allows the crystallizer to be designed with the necessary impeller, baffles, and other fixtures to optimize the crystallization operation.

Other Tips – Crystallization is as much art as science, and many approaches have been investigated to circumvent problems. For example, cycling the slurry temperature up and down can increase particle size and reduce fines. Aging the slurry can sometimes do the same. Polymorph shifts can be induced by slurrying the crystals in a non-solvent at just below the melting point. This is sometimes carried out on seed material, to ensure that it consists as much as possible of the most stable polymorph. The addition of co-solvents should be tested at various temperatures. Sometimes it is advantageous to perform the addition at a high temperature, and then cool the whole mix. In other cases, better results are obtained by cooling first, and then adding the second solvent. Pay attention to the possible effect of water on the crystallization. Large-scale phase separations may not be as clean as at the bench, and excess water may have to be removed by azeotropic distillation. Ensure that vigorous mixing is used when samples are collected for water analysis.

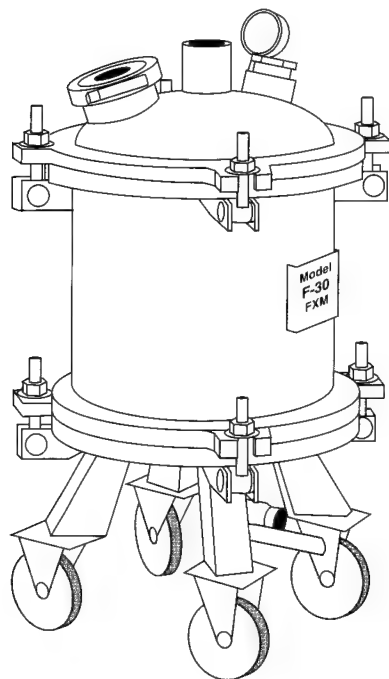
Product Isolation

Product isolation here refers to the separation of crystalline or other solid products from a solid-liquid slurry by some means of filtration. The filtrate, the liquid that passes through the filter, is often called the product liquors or “mother liquors”, while the solids collected in the filter are called the “filter cake”. Because filtration behavior depends very much on solids properties, particle size analysis of the crystalline slurry is vital to the development and optimization of filtration processes and equipment selection. Crystallization and isolation processes should be scaled up hand-in-hand to ensure that a consistent filtration feed stock is supplied every step of the way.

Cake filters range from simple types, which resemble the common laboratory Buchner funnels, to highly specialized automated equipment, such as centrifuges, Rosenmund-type jacketed filter-dryers, and continuous rotating-drum vacuum filters. Small versions of some of these systems are available for pilot-scale use, and in many cases, such units are the best tools for predicting the performance of their manufacturing-scale counterparts. But for flexibility, ease of use, low cost, and a minimum of cleaning and maintenance requirements, a simple Nutche-style pressure filter can serve quite well in small pilot facilities. They are manufactured in many materials and sizes up to several feet in diameter.

Vacuum and Pressure Filters – The simplest batch filters are the Nutche-style filters, which are usually operated by vacuum or by positive pressure above the cake. They are very useful at the small pilot scale, but with increasing size become more difficult to operate. Usually, the product must be discharge manually, which makes them less desirable for production use. These filters are little more than vessels with a perforated bottom on which the filter cloth sits. They may be jacketed to accommodate hot or cold filtrations. The simplest ones are not covered, which means there is no protection from exposure to the solvent vapors or the product itself. The covered style are safer for many obvious reasons if flammable or hazardous substances are in use. They can better protect the product from drying out before wash is applied and can also be pressurized above the cake to increase filtration driving force. Some have agitators to stir up the filter cake and provide better distribution of wash. The figure below shows a typical pilot scale pressure filter. Note that any vessels that will be pressurized must be pressure-rated as per ASME Code.

Operating Tips for Small Pilot Filters – To ensure safe operation and repeatable results, it is a good idea to have a detailed written operating procedure that can be used for training and be referred to during actual operations. Sections should include setup (assembly, pressure check, proper grounding, inerting, vacuum cold trap preparation), operation, product discharge, disassembly and cleaning. Pressure gauges or other sensing devices must be periodically calibrated. Always double check the materials of seals and gaskets to ensure their compatibility with the chemicals in use. Use PTFE-lined hoses of sufficient diameter, and avoid constrictions to ensure that the slurry will flow.



A Typical Non-Jacketed Pressure Filter

Continue mixing the slurry as long as possible during the filtration. To ensure even cake distribution, fill the filter about halfway with slurry before applying the vacuum or pressure. Do not allow the cake to completely drain before adding more slurry or it may form cracks. Monitor the progress of the filtration by recording mother liquor amount collected over time. As the level drops in the crystallizer, reduce agitator speed to prevent splashing and vortexing. If a heel is left in the vessel, circulate the mother liquors back to wash it out.

Avoid letting the cake become too dry before applying the wash. If the cake cracks or separates from the sides, it should be smoothed out before the wash is applied. If it is not possible to see the state of the cake, the filter should be opened to check it, because a cracked cake will not wash effectively. Be careful to minimize exposure to the air if it is an air or moisture-sensitive compound. A good general rule is to use two cake-volumes of wash, applied in two portions. After washing, drain the cake as much as possible by applying an inert-gas blanket over the cake.

Digging the solid cake out of the apparatus can be dangerous because of the

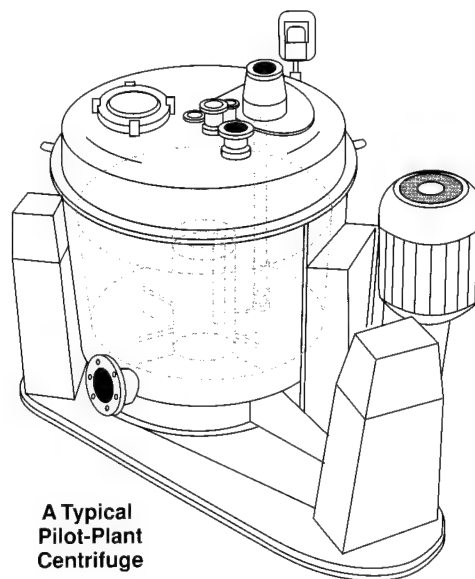
strong possibility that the discharge of built-up static electricity could ignite the powder or any solvent vapors that may be present. Proper grounding is critical, but the filter must also be fully inerted with nitrogen or other inert gas.

Filter-Dryers – Rosenmund and 3V Cogeim are two well-known manufacturers of a highly specialized type of Nutche filter designed for large scale operation, with filter areas up to 100 square feet or larger. They usually include mechanized wash arms, cake spreader, and discharge screws for totally enclosed operation. Pilot-scale filter-dryers are available. These are expensive but ideal for minimizing product handling and exposure where contamination could compromise quality, or for water-reactive or air-reactive compounds. However, they usually offer relatively small surface area for their cost, and can introduce other problems such as the generation of heat or static from friction and crystal breakage while the cake is being agitated.

Product Centrifuges – Centrifuges offer a number of advantages for product isolation compared to simple vacuum or pressure filters. They can generally spin more solvent out of a cake (they typically operate at centrifugal forces about 1000 times gravity), which can reduce drying time. Many are bottom-discharge units with mechanical ploughing systems that greatly speed up processing of multiple loads, allowing much higher throughput than simple pressure filters. Such discharge systems can greatly minimize worker exposure to the wet cake. Centrifuges are typically used when solids content of the slurry is greater than 10% and the product is of relatively large particle size and the cake is incompressible. Thus, not all materials are suitable for use in centrifuges. The tests described on page 2-27 can be helpful in determining if a centrifuge should be considered for a particular compound. The choice of filter cloth is also critical to successful operation. Cloth types are discussed in more detail on page 2-26.

A centrifuge should be selected only after tests have been performed on similar equipment using product slurries identical to those that will be experienced at scale. Bench measurements can help narrow down the field, but are not a substitute for working tests with laboratory scale centrifuges or pilot studies. Any reputable manufacturer of centrifuges can perform tests for you at their facility or at your own in certain cases.

The centrifuge usually consists of a horizontal or vertical spinning basket, lined with filter cloth or other filter medium, a feed nozzle that directs the slurry gently towards the wall of the spinning basket, a discharge port through which the liquors can be removed, and a bottom discharge hatch through which the product cake can be collected. On smaller models the product must be discharge manually out the top. The figure at right shows a typical pilot scale product centrifuge. Because these units operate at high speeds, the motor/basket assembly must be securely mounted to prevent damage in case of imbalanced loads.



**A Typical
Pilot-Plant
Centrifuge**

Centrifuges usually employ explosion-proof motors for handling product slurries in flammable solvents, but most facilities also require that they be fitted with automatic purge-interlock systems that will not allow the centrifuge to be started if the oxygen concentration is above a certain minimum value. Other interlocks prevent opening the centrifuge when it is in operation, or disconnect power when the unit is off-balance because of uneven loading. Centrifuges are relatively simple mechanical devices, but operate at high speeds and are sometimes subject to intense vibrations, which puts great strain on bearings, etc. This, along with the interlock systems can make them rather high-maintenance units. Some major manufacturers of product centrifuge equipment include Sanborn, Westfalia, Heinkel and Western States Machine, and Broadbent.

Operating tips for product centrifuges – Proper operation of a product centrifuge requires training, experience and an attention to detail. Much of the operation depends on the nature of the product and the slurry, but certain basic principles should always be observed. During setup, the loading nozzles should be positioned in such a way that cake will be distributed evenly over the sides of the basket. If the nozzle is too low, a “heel” of product will accumulate in the bottom of the basket. If too high, product slurry may splash over the sides of the basket. Proper nozzle placement is likewise

important for efficient cake washing. Proper fitting and seating of the cloth is also critical. Feed pumps and lines should have no constrictions and be large enough to provide sufficient flow of the slurry. The outlet should be sized to prevent buildup of liquors in the unit, which can put an excessive load on the motor. The liquor collection tank must be vented to prevent pressure buildup.

During product feeding, the slurry should be kept uniform by agitation and the centrifuge should be operated at slow to medium speed. But there the science of centrifuge loading gives way to “art”. Feeding too quickly can cause flooding and product loss. This can be corrected by decreasing feed rate, increasing centrifuge speed or by using a more porous cloth. On the other hand, feeding too slowly will allow the cake to dry out before it is evenly distributed, causing an imbalance in the basket. This can be corrected by using a more dilute slurry, decreasing centrifuge speed, by feeding more quickly or by using a less porous cloth. Do not overfill the centrifuge. If the slurry level falls below the bottom of the agitator and begins to settle before it can all be loaded on the centrifuge, a significant heel can be left in the vessel. This can be difficult to remove. One approach is to circulate the liquors from the last load back to the vessel through a coarse spray nozzle or the like to ensure that all of the product is washed out to the centrifuge.

Once the cake is loaded, the speed can be increased to “spin out” the cake to remove as much of the liquor as possible. However, care must be taken not to allow the cake to crack if a solvent wash will be applied to displace the liquors. A crack can cause inefficient washing. If a crack appears, the centrifuge should be stopped and the cake smoothed out before washing. The final spin-out can be delayed until all of the washing has been completed. The wash feed rate must be controlled in similar fashion to the product feed rate. Feeding wash too slowly will not wash the entire cake uniformly. Feeding too quickly can flood the bowl or bore holes in the cake. After the final high-speed spin-out, slow the centrifuge back down to minimum speed for ploughing out the cake, or stop it for manual discharge.

Filter Aids – Filter aids are non-compressible particulate substances that are either mixed with the product slurry to improve filtration rate and reduce cloth blinding, or laid down to pre-coat the filter before the slurry is introduced to improve retention of fine particles. The most common filter aid is Celite (diatomaceous earth). The obvious disadvantage of mixing a filter aid with the product is that it has to be removed later, which may or may not be a major concern depending on the process.

Filter Cloth – The filtering element in most filters or centrifuges is a type of cloth that can be woven or nonwoven (felts) and made of any number of materials such as cotton, polyester, nylon, polypropylene, etc. Even fine-mesh cloths of stainless steel, Hastelloy and other metals are available where greater strength is required. However, the surface area of such woven metallic cloths is limited to the open geometric surface area, whereas nonwoven felts act as “depth filters”, meaning that pores deep in the cloth will collect product, giving it a greater effective surface area. Clothes are often classified according to porosity (measured in microns) and permeability (ft³ air/min, or CFM).

In reality, the filter cloth often acts as no more than a substrate for building up the first thin layers of the filter cake, which itself then acts a filter to trap more and more particles of smaller and smaller size. Thus, the first liquors to come through a filter are often not crystal clear due to fines passing through the cloth. Once the initial cake is deposited, the liquors clear up. In some operations, the liquors are circulated back to the slurry tank until it is determined that they are clear, then they are directed to a liquor collection tank. In this way, no product is lost to “breakthrough”. Naturally, the pressure drop through the filter cake increases and flow decreases as the filter cake gets thicker. If the slurry contains too many fines, all pores of the cloth may become blocked. In this case the cloth is said to be “blinded” and no liquid will pass through.

Cloth filter media are widely used because they are relatively inexpensive. But because of the great variety available, selecting the optimum cloth for a given application requires some thought. The wrong cloth can hurt the entire operation. Yields and cycle times will suffer, costing a great deal in the long run.

Woven fabrics are a very common choice for filtration media because they are strong and available with a wide variety of characteristics. Weaves are usually of the plain one-over-one-under **basket weave** (very tight), the **twill weave** (medium porosity) or the **satin weave** (most open). Other available weaves are **duck** and **chain**. Where **monofilament**-fiber weaves are applicable (usually for coarse crystalline products with large particle sizes) they are advantageous because they do not blind easily and product is easily removed. **Multifilament** yarn weaves are capable of retaining

much smaller particles, while spun-staple yarns act as depth filters and have the highest fines retentions. The fiber **ply** refers to the number of individual fibers twisted together to make a single strand of yarn. The **count** refers to the number of threads per inch. Woven fabrics are also finish-treated by **calendering** (hot rolling), **napping** (fuzzing the surface) or **heat setting**, all of which affect the porosity and retention characteristics and the blinding tendency.

Filter cloth selection can be simplified if sufficient data exist about the nature of the application. Useful information about the process includes: size, density, and crystal shape of the product, the solids content and uniformity of the slurry, settling time, cake compressibility, desired cake moisture content, batch temperature, pH and chemical composition. These must be compared to the pore size, material and pressure susceptibility of the cloth. Any reputable filter media or filtration equipment supplier can provide samples of various cloths that meet your criteria for you to test. Bench-scale tests should be carried out by using samples of the material in a number of repeat filtrations. If the resistance increases after several filtrations, it is a sign that the cake is becoming clogged or blinded and may not be a good choice for operations such as multiple-load centrifuge batches. Sefar, Katema and Crosible are reputable filter cloth manufacturers.

Filtration Scale-Up

Most fine-chemical and pharmaceutical products are solids, typically isolated from a slurry. Thus, filtration is an important operation that can significantly affect product cost. Slow filtrations can become the rate limiting step in a process cycle and high solvent content can drastically increase drying times. Thus, it is useful to try and understand the factors that can impact the success of large-scale filtration and the selection of isolation equipment.

Lab-bench filtrations are not particularly representative of larger scale isolation equipment, but there are some simple preliminary tests that can help determine the prospects for good filtration on a pressure filter or centrifuge.

First, a simple **settling test**. Allow about 1 liter of slurry to settle on the lab bench in a beaker; the material should settle and produce a clear liquor phase in well under 30 minutes. If the slurry remains cloudy for 30 minutes or more, the product will probably be difficult to isolate. The crystallization conditions may have to be altered to increase particle size or reduce fines.

Second, a **cake permeability test**. Filter a slurry sample through a buchner funnel with vacuum to obtain a filter cake about 2" thick. Then, measure the rate at which clear mother liquors can be filtered through the cake (make sure there are no cracks in the cake). If the liquors filter at a rate of 1 gpm/ft² (40 lpm/m²) of filter area or greater, then it is a good candidate for centrifugation or other large-scale filtration. If the liquor filtration rate is less than 0.5 gpm/ft² (20 lpm/m²), it means that the slurry contains too many fines, or that the product is amorphous (noncrystalline) in nature and too easily compressed to allow liquid to drain through. Again, a change in crystallization conditions may be required.

Pressure-Filter Tests – More detailed tests can be performed using a pressure-filter test apparatus. Filtration is practiced in many modes, but the most common for product isolation is *constant-pressure* mode, in which the driving force, be it positive pressure, vacuum, or centrifugal force, is held constant throughout the cycle. The filtration rate typically decreases as the cake builds up, and can sometimes stop altogether if the cloth becomes blinded or clogged with solids.

In a common laboratory method, a slurry sample is vacuum- or pressure-filtered through a Buchner or similar funnel designed for the purpose. The volume or weight of filtrate collected is measured over time, and these time/filtrate data are plotted in one of several ways for linear regression analysis. The slope of the straight line obtained is used to derive the desired parameters characteristic of the slurry. The slope is also a function of operating pressure and thus it is useful to use a test pressure similar to that expected for large-scale operation. See the example on the following page. It is important to use good experimental technique when collecting the data. The slurry should be kept as homogeneous as possible throughout the test, and data points obtained after the cake begins to drain are not valid.

Two common equations used for these calculations are the so-called Tiller equation and that offered by Strauss in [242], both of which are shown in the example on the following page. The Tiller equation enables calculation of the **specific cake resistance**, α , a classical measure of filterability, which can range from 10⁹ m/kg for easily filtered materials to >10¹³ m/kg for difficult gelatinous or amorphous solids. The Strauss equation may be somewhat simpler to use. The value of the slope obtained, m , is only a relative number, and should be compared with values for known compounds,

Constant-Pressure Filtration Test Example

The following data were collected during a filtration test using a 5 in² vacuum filter (assume $\Delta P = 14.7$ psi):

Time (min)	Wt. Filtrate		Calculated Values	
	(g)	(lbs)	V/A	Pt/(V/A)
2	43	0.095	0.0190	1547
5	94	0.207	0.0414	1775
8	137	0.302	0.0604	1947
14	207	0.456	0.0912	2257

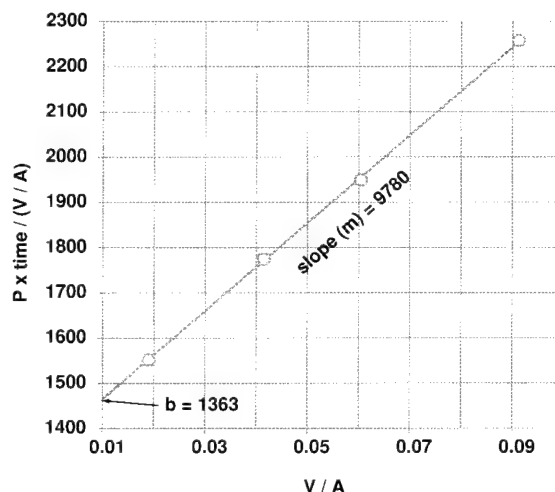
The data can be plotted using the equation suggested by Strauss [242] as shown at right to obtain slope m , a relative measure of filtration speed (see text):

$$\frac{P \times \text{time}}{V/A} = m \frac{V}{A} + b$$

The other relationship commonly used is the Tiller equation:

$$\text{time} = \frac{\mu \alpha c V^2}{2A^2 P} + \frac{\mu R_m V}{AP}$$

In this case, a plot of time/volume vs. volume generates a straight line with a slope of $\frac{\mu \alpha c}{2A^2 P}$ from which α , the specific cake resistance, can be derived. In this equation, μ = viscosity, c = solids concentration, R_m is the resistance of the filter medium, which is constant. For more details on the use of this and other filtration relationships, see references [59, 169, 202, 233, 243].



but it can give a good indication of the likely success of the filtration on scale up. Also, armed with values for the slope and intercept, useful predictions of scale-up filtration time can be made by substituting plant values of A and V . It doesn't matter what units are used as long as they are used consistently. When pressure is expressed in psia, area in in², filtrate in lbs, and time in minutes, the Strauss equation gives values of m that range between 1000 for free-filtering materials, to >400,000 for very difficult materials. If tests are run at several different pressures, the slopes of the lines obtained by regression analysis should be identical; i.e. the lines should be parallel. If not, it is an indication that the cake is compressible.

This discussion is only intended to enlighten the reader as to the potential utility of simple bench tests. There are many other approaches to using the same fundamental bench data. These are described in detail in a number of excellent sources [56, 169, 202, 233, 243].

Final Tips for Pilot Filtrations – Before isolating the product, collect slurry samples to be sure that the crystallization has reached equilibrium. It may take longer for all of the solute to crystallize at larger scale. Rather than risk a low yield, filter a sample and determine the concentration of product in the mother liquors, either by HPLC or by evaporating and weighing it. Another suggestion well worth the effort: retain a sample of the pooled mother liquors/wash and record their total weight to simplify troubleshooting later when the yield *still* comes out low. Better yet, save the entire mother liquors and wash in the receiver or *clean* drums until a dry weight on the product is obtained.

If the isolation temperature is very low, or very high, the thermal mass of the room-temperature isolation equipment may have a significant affect on solubility, yield and purity. It may be necessary to precool or preheat the isolation train to prevent a low yield or prevent product from crashing out. Also ensure that the isolation equipment is completely dry, especially if the last solvent used to clean it can interfere with the isolation. A liter of methanol left in the bottom of a centrifuge could dissolve half your product, especially if the mother liquors are to be cycled back to the crystallizer to remove the product "heel". It may be wise to flush the equipment train with solvent of the same composition as the mother liquors prior to isolation.

Product Drying

Drying is an important operation for the production of consistent, stable, free-flowing solids for packaging, storage and transport. But as with product isolation, the drying operation should not be considered as an isolated step. It is best viewed as part of an integrated process that includes crystallization and isolation, since changes in these operations that affect particle size range and moisture content, can have a significant impact on the efficiency of drying.

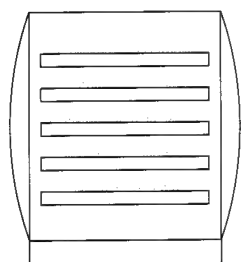
Drying is not a particularly energy efficient process. Consider, for example, that it can take 5-10 times the amount of energy to remove a kg of solvent in a drying operation than in a distillation operation. Consequently it is important to remove as much solvent or moisture from the cake as possible beforehand. The choice of isolation method is important in that regard – centrifuges typically produce the driest cakes, followed in turn by pressure filters and then vacuum filters. But the choice of isolation equipment ultimately depends on the physical characteristics of the product.

Drying Requirements – Before approaching any drying operation, it is necessary to know what the requirements are. Drying to “zero” moisture or solvent content is not practical. Therefore, the first step is to set a realistic drying specification consistent with good product stability and handling characteristics. The specification is, of course, test-dependent. For crystalline solvates, for example, a loss-on-drying (LOD) test will give very low values, whereas TGA or GC will give much higher results. The LOD is more indicative of the success or completeness of the drying operation, since it measures the amount of unbound solvent or moisture.

Equipment Classification – There are a great many types of dryers. They are usually classified according to the mode of operation (batch or continuous) and the method of heating. In *convective dryers*, heated air or gas is passed over or through the cake material. These include fluidized bed, spray, gravity and convective tray dryers, among others. These types of units tend to keep the product relatively cool, but they are not very energy efficient, and often require more elaborate dust collection, solvent recovery and gas recirculation systems. They can be scaled up to very large sizes and are usually run in continuous mode, often for dedicated products, but can also be used quite successfully in batch mode. Fluidized bed drying lends itself well to batch mode operation, as does spray drying. Spray drying is an interesting technology that combines three unit operations (crystallization, isolation and drying) into one. It is especially useful for compounds that cannot be crystallized by conventional means. However, since no purification takes place, feedstocks for spray drying must be very pure. Also, cleaning the units between batches or products can require considerable effort.

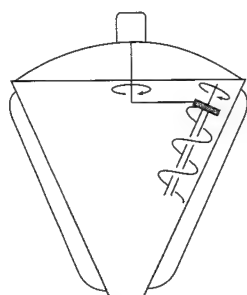
In *conductive*, or *contact* dryers, the cake material directly contacts the heated dryer surface. These types include the smaller, batch-style dryers most often found in pilot plants, such as tray, rotary cone, paddle, and tumble dryers. A carrier gas is often used with these types as well, not to impart heat, but to carry off the evaporated liquid. Other heating mechanisms, used mostly for specialty applications, include microwaves, irradiation and dielectric drying. Perry [195] offers a very comprehensive survey of industrial drying equipment and principles. The remainder of the discussion here will focus on batch, conductive drying.

Common Types of Contact Drying Equipment



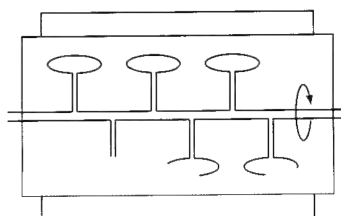
Vacuum Tray Dryer

Simple, labor intensive,
long drying times.
Encrustation/poor uniformity.
Post-treatment often required.



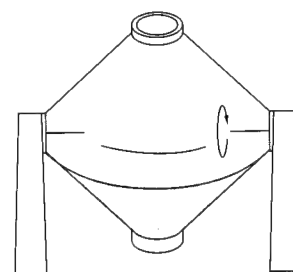
**Rotary Cone Dryer
(orbiting screw)**

Active agitation,
good homogeneity, but
high particle attrition.



Paddle Dryer

Medium agitation,
good homogeneity,
less attrition.



**Tumble Dryer
(rotating double cone)**

Gentle agitation,
good product
homogeneity.

Drying is often carried out at reduced pressure. This is particularly effective for temperature- or air-sensitive products and safer for drying toxic substances and those containing flammable solvents. Small units are usually heated electrically, but for increased safety in larger units, steam or other heat transfer medium is circulated through the dryer trays or unit jacket. Low-pressure steam can provide temperatures up to about 150°C. For higher temperatures, synthetic heat transfer fluids are used (for more on heat transfer media, see Chapter 4).

Equipment Selection – The most common dryer for laboratory and small-scale pilot work is the vacuum tray dryer. It can be scaled up to moderate size, but space and efficiency limitations make further scale-up impractical. It is reliable and has no moving parts to fail, but operation and cleaning are quite labor intensive. Also, because tray drying can cause a crust to form on the cake, the product often requires milling, screening, blending or some other type of post-drying treatment to ensure homogeneity. Problems with encrustation and non-homogeneity can be circumvented by keeping the cake moving during drying. This is the major advantage of cone, paddle and tumble dryers and agitated combination filter-dryers. Cake movement can also reduce cycle time, depending on the nature of the solvent in the cake.

As always, it is important to understand your operational needs and capacity requirements before selecting drying equipment. Work only with reputable vendors. They can provide a wealth of information about the advantages and disadvantages of various units for your application. Do not overlook ease of cleaning. This is particularly critical in multiuse pilot equipment and GMP environments.

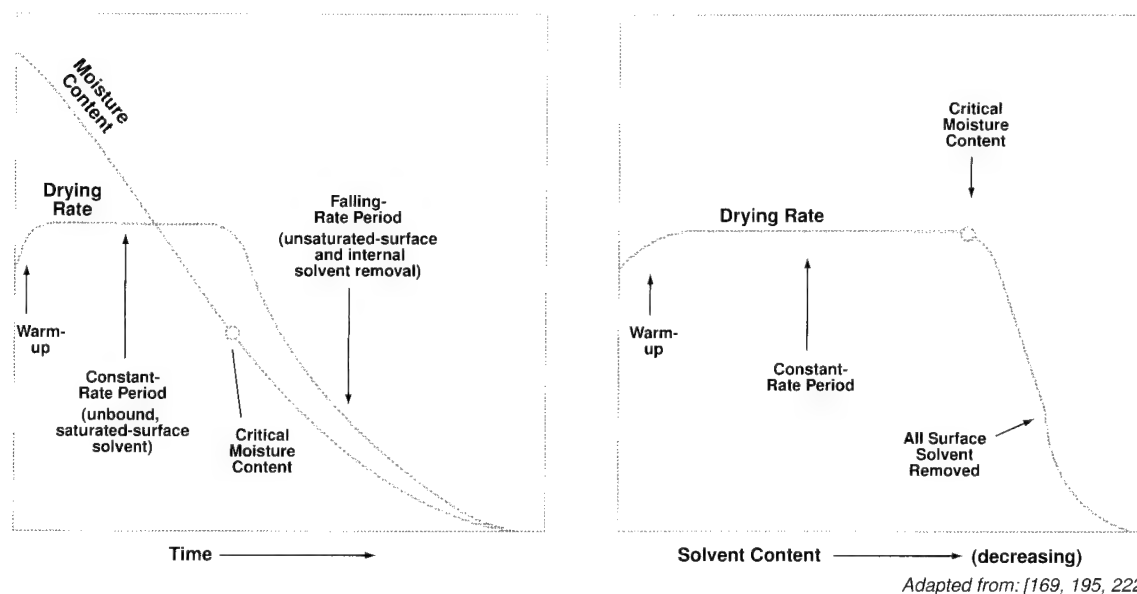
Product Characteristics – The properties of product from pilot drying equipment may be significantly different from that dried in laboratory vacuum tray driers. This is particularly true of units that agitate the cake mechanically such as orbiting screw conical dryers. Particle attrition or agglomeration can result in major differences in particle size distribution, bulk density, compaction and flowability. These things in turn affect solubility, bioavailability, formulation processing, packaging and shipping. Therefore, it is not valid to base projected product properties on the results of tray dried samples when different equipment will be used on scale-up. The behavior of a given product in different dryer types cannot be easily predicted. Bench or small pilot-sized test units are available for tumble or paddle driers, but the dynamic similarity to large-scale equipment is poor. The best way to determine what the product will look like is by performing pilot studies in representative drying equipment. Sometimes the actual product characteristics will not be known until the first production batch comes out of the dryer.

Predicting Drying Cycle – As mentioned previously, predicting the physical characteristics of dried products is not often very successful, but it is possible to estimate the approximate drying cycle time on scale-up. The first step in making such a prediction is to determine the major resistances to drying. This involves performing a simple vacuum oven test, during which a sample is dried and data on solvent content vs. time is collected. For completeness, the test should be performed on several samples of varying cake thickness. For the test to be most meaningful, the sample used must be representative of the final process material.

Several types of drying may occur during a drying cycle. As the conditions in the dryer approach the boiling point of the solvent, the first solvent to be removed is that which is free, unbound and saturating the surface of the solids. This is removed at a fairly constant rate until the surface is no longer saturated. Once the surface solvent is removed, then solvent that is trapped in interstitial spaces and microcapillaries is removed. Evaporation of this solvent is slower because additional energy is required to overcome capillary attractive forces. Next solvent that is completely trapped in vacuoles may be removed, but the rate of removal is very slow, limited by diffusion. It is best to assume that this solvent will not be completely removed, nor will solvent that is part of the molecular crystal lattice.

The drying test mentioned above can give a good indication of the major resistances to drying. The solvent/time data can be plotted directly, but it is more useful plotted as *drying rate vs. time*, or as *drying rate vs. solvent content*. Examples of such plots are shown on the following page. The period where the surface moisture is removed is called the constant rate period, up to the point where there is no longer sufficient solvent to make a continuous layer over the surface (called the critical moisture content). This is followed by the falling rate period, characterized by an ever-changing drying rate as, first, the solvent from the unsaturated surface is removed, followed in turn by the various components of the internal solvent. Often the falling-rate period dominates the drying time requirements. It is a case of diminishing returns, which is why a reasonable drying specification is so important. At any one time, several drying mechanisms may be happening in parallel, but one mechanism usually dominates.

The Stages and Major Resistances in Product Drying



Many actual drying curves may not appear to fit this model well. Some product wet cakes may come out of the filter already below the critical moisture content, and then the entire drying cycle will consist of falling-rate drying. This is one reason why it is valuable to know the critical moisture content. In other cases, a short period of rapid solvent removal may occur, followed by a settling-in to the falling rate period. This is most likely to occur if a product cake is placed in a preheated dryer, and then the vacuum applied.

The actual calculations for predicting cycle time in various drying units are rather rigorous. The total drying cycle is the sum of the constant rate and falling rate periods. Calculating the falling rate period is difficult because of the effect of various diffusion coefficients, etc. Calculating the constant rate is somewhat more straightforward, but requires knowledge of the following parameters, among others:

- amount of solvent to be removed (wet batch weight – dry batch weight)
- heat transfer area of the unit and overall heat transfer coefficient
- solvent enthalpy of vaporization
- temperature difference between the jacket and the evaporation temperature at the drying conditions
- solids density and cake thickness

Details of the calculations are not presented here. For a more in depth study, see [169, 195]. Drying equipment manufacturers can also help you estimate cycle time, based on the results of your drying studies. In reality, however, because there is so much variability, estimates can be off by 50% or more. That is why there is no substitute for completing pilot scale studies in representative equipment.

Even without completing the rigorous calculations, the results of the bench studies can provide a great deal of useful information. For example, if the constant rate period predominates in the bench test, then it will likely predominate at scale. Agitated dryers may then offer the shortest drying times by increasing the effective surface area of the cake exposed for heating and drying. Tray dryers may not be so advantageous since increased cake thickness in larger-scale units means decreased heating surface area per unit mass.

If the falling rate period predominates, it means that the process is diffusion limited and this period will likely predominate at scale. In such cases, agitated dryers such as orbiting screw cones or combination filter dryers may offer advantages by increasing the surface area for diffusion by particle attrition.

Energy Requirements – The energy required for batch drying can be estimated based on the amount of solvent to be removed (cake wet weight – cake dry weight), and its enthalpy of vaporization. For a first pass estimate, sensible heat can be ignored.

Tips for Operating Small Dryers – Most of the suggestions here deal with vacuum tray driers, but many are applicable to the operation of other types of drying equipment as well. As with any operation, it is important to have a start-up checklist to ensure that no important detail is overlooked in starting a drying cycle. Also, prepare detailed cleaning procedures and establish cleaning criteria (see page 2-34). Record all operations, including cleaning and maintenance, in the equipment log book. The diagram at the bottom of the page shows a typical setup for a vacuum tray dryer.

Ensure chemical compatibility of seals and gaskets with the solvents in use and the compatibility of the tray and dryer materials with the product. Make sure that no corrosive vapors will attack dryer surfaces. Polyethylene tray liners can be used if desired, depending on the drying temperature. Make sure that the solvent trap is empty and operational. Cryogenically cooled traps can be a convenient substitute for dry-ice/alcohol traps, but make sure that the heat removal capacity will match the peak solvent removal rate. To ensure sufficient capacity, determine roughly the amount of solvent to be trapped, or plan on monitoring the solvent trap level.

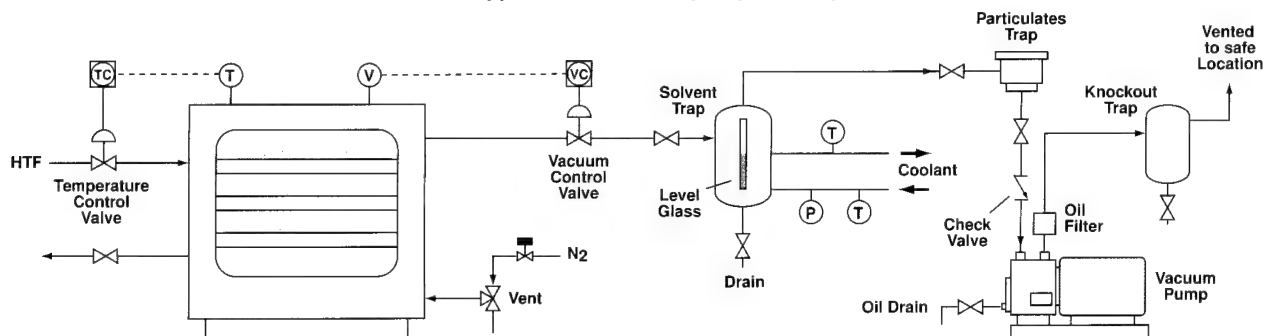
Lines and connections must offer no restrictions that could be clogged with ice or other crystals (often a finite amount of water is removed from cakes wet with even hydrophobic solvents). Include a dust trap upstream of the vacuum supply and a solvent knockout trap at the vacuum exhaust. Include an inert gas supply line to release vacuum, especially for air sensitive or hygroscopic compounds. It is also common to leave a slight nitrogen bleed on during drying which increases convective removal of evaporated solvent, but if the gas flow rate is too high, uncondensed vapors can pass through the solvent trap. The nitrogen bleed also works well to prevent condensation buildup on the dryer window glass.

If covered drying trays are used, make sure to leave an opening to allow unrestricted removal of the solvent. Place the product in the dryer, apply vacuum, then apply heat. This can help prevent a bolus of solvent from overwhelming the solvent trap condenser as vacuum climbs.

The temperature stability of the product must be considered in setting the operating conditions to ensure that it will not be heated above the allowable limit. Generally speaking, the higher the vacuum the better, in order to keep evaporation temperature low and maximize the temperature difference with dryer surfaces. On the other hand, a certain minimum temperature may be necessary to reach the specification. Some compounds undergo polymorphic shifts well below their melting points, and this needs to be understood as well. It can be useful to monitor the product temperature during the drying cycle. When the temperature stops rising and levels off, it's a good indication that drying is complete.

Vacuum pump oil can quickly become contaminated with solvent or water, reducing pump efficiency. Check the oil level frequently, and change it when it begins to get too high due to absorbed solvent. It's a good idea to drain and change the oil between batches. Pump oil is inexpensive when compared to the value of the product. To simplify this sometimes messy task, set up a drain valve to make draining the oil more convenient. For more on vacuum pump maintenance, see page 7-22.

Typical Vacuum Tray Dryer Setup



Scrubbers

The purpose of a scrubber is to prevent noxious or hazardous gas components and dusts from escaping into the atmosphere by absorbing them in a liquid stream for disposal. Scrubbers usually consist of a packed tower or slotted-tray column designed to maximize gas/liquid contact area for most efficient extraction of pollutants. Performance is limited by thermodynamic adsorption equilibria. The gas stream is often injected into the bottom of the unit; the liquid is sprayed from the top and allowed to trickle down, stripping out the gas as it falls. This is called countercurrent flow. The basic components and operating principle behind most industrial scrubbers is shown in the diagram below.

Proper design of a scrubber is quite complex and involves a good understanding of mass transfer, Henry's law, gas/liquid diffusivities, etc. Performance must be maximized while keeping the unit size as small as practical and minimizing foaming and other operational problems. Chemical compatibility of components is critical. An in-line heat exchanger will allow temperature control should it be necessary to remove reaction heat or to warm the solution to prevent freezing of outside units or to improve extraction efficiency. For any scrubber of appreciable size, environmental permitting will no doubt be required.

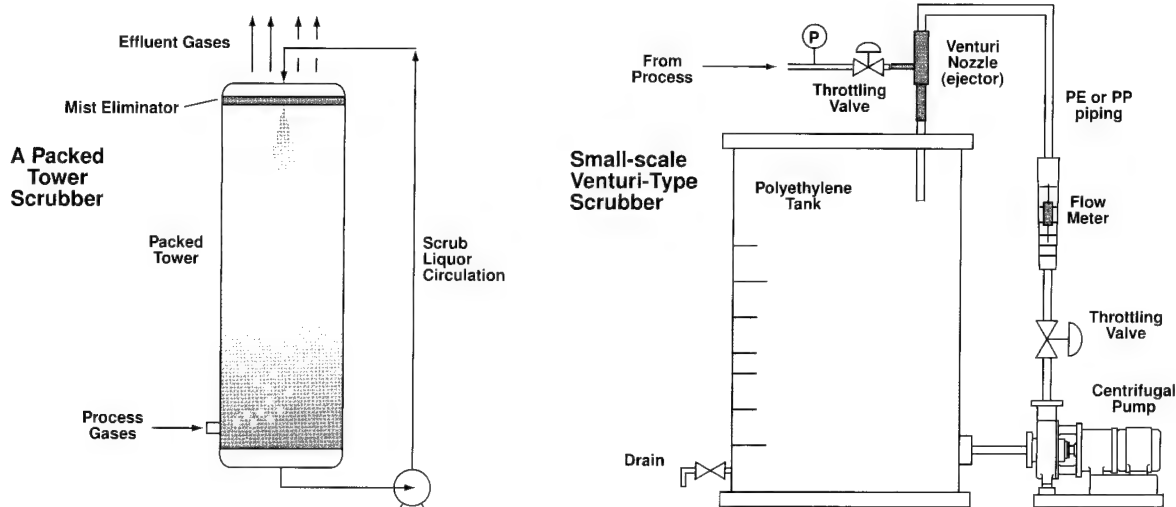
Small commercial scrubbers are available from many suppliers of chemical reactors and pumping equipment. Work only with experienced manufacturers, and be able to provide estimates of the required capacity and data on the types of substances that will be used. A small venturi-type scrubber, such as shown below, can be constructed for kilo-lab use, but it is important to understand its capacity, to use the same care in selecting the scrub solution as with any larger unit, and to operate it only within a certified walk-in chemical fume hood.

Scrub Solutions – Selection of the scrubbing solution should be based on laboratory data or previous experience. A few of the solutions commonly used are listed here, but none of these should be used without testing its effectiveness in your particular circumstances:

- Acidic vapors (HCl, HF, SiF₄, SO₂, Cl₂, CO₂, HCN) – plain water or alkaline solutions such as dilute NaOH, K₂CO₃, ammonia water, or ammonium salt solutions.
- Sulfurous (H₂S) and other noxious fumes – NaOCl solution
- Odorous and organic compounds, Br₂ – oxidizing solutions such as KMnO₄, HNO₃, H₂O₂, NaOCl, Na₂S₂O₅
- Organic vapors – high-boiling oils or solvents, but volatility must be minimized to prevent oxidation and fire.

Analytical tests must also be available to determine when a scrubbing solution becomes exhausted. Often a simple pH measurement can give a good indication of the remaining solution capacity.

Maintenance – The scrubber can become a breeding ground for bacteria, and will often accumulate mineral deposits that can minimize scrubbing efficiency. It must be periodically drained and flooded with hypochlorite, dilute acid or other cleaning solutions as appropriate. Mist eliminators can plug and need to be serviced as well.



Reactor and Equipment Cleaning

Ensuring that equipment is clean between batches and between products not only makes good engineering and business sense, but validated cleaning procedures are required in any processes involving human pharmaceutical products. Proper cleaning is an involved process that must consist of at least the following four major components:

- Identification of the possible contaminants – these might include the major product itself, intermediates or raw materials, side products or impurities, or cleaning agents and detergents.
- Establishment of the maximum acceptable residual amount of contaminant for the particular equipment. For multiuse equipment, this means specifying the maximum allowable carry-over of one product into another. This is obviously set very low, say 10 ppm, but depends on the activity of the compounds involved. Calculations are then developed, based on the surface area of the equipment, expected amount of material that may adhere to equipment walls, and typical batch size, to determine a maximum allowable concentration in final rinse solutions that is consistent with the acceptable carry-over. Be conservative in your assumptions. [100, 270] are good reference for more information on this topic. Manual inspection to ensure that no visible residue remains in the equipment must also be part of the cleaning criteria.
- Development of cleaning solutions and cleaning procedures validated to prove that they effectively remove the expected contaminants or detergents. This includes identifying recommended cleaning solvents or solutions and establishing that the solubility of expected contaminants is high enough to readily dissolve any residual left in the equipment. The use of detergents should be considered carefully to ensure FDA acceptance for drug manufacture.
- Development of analytical test methods, validated to prove that they can detect the expected contaminants or detergents with sufficient sensitivity to ensure their removal to levels below the allowable limit. This must include system suitability tests and the certification of appropriate reference standards. Cleaning procedures and tests should be product-specific. For detergents, analytical test methods may be available from the manufacturer.

A typical cleaning procedure may start by flushing out the equipment with a pressurized water spray or similar initial rinse to remove gross contaminants. This would be followed by cleaning with solvent, detergent, or rinse solutions, which are either refluxed in the equipment or circulated through the spray ball or equipment train for a specified time. This should be repeated a sufficient number of times, and with whatever combination of solutions is deemed necessary to ensure that the equipment is clean. Then a final rinse solution, using a specified volume of a solvent in which the contaminants are highly soluble, is circulated through the equipment and sampled for analysis.

A unit should be released as clean only after it passes visual inspection and the criteria of the analytical test procedures. Cleaning procedures and results should be documented in detail and become part of the equipment's permanent record. Each piece of equipment should be labeled to indicate its state of readiness – "Cleaned", "To Be Cleaned", etc.

Cleaning operations should be described in detail in approved, written SOP's. All aspects of the operation should be specified, including solutions to be used, operating temperatures and pressures, key valve positions, etc. The format should be similar to that for batch records (see page 1-14). Operators must be fully trained in their use.

Cleaning should be considered before new equipment is purchased. Plans can then be made to include a spray ball or other automated cleaning system that will reduce the labor associated with manual cleaning. Even with a spray ball, however, some components may need to be removed or disassembled for manual cleaning, inspection, or swab testing.

Units may be cleaned individually, but it is not uncommon to clean an entire equipment train by circulating cleaning solutions through all the individual units connected by hoses. More thought must be given to this approach since the entire train will fail clean testing if one unit is dirty, but the advantage is that fewer samples need to be collected and everything, down to individual hoses, can be tested and released in one fell swoop. Beware of holdup in dead-legs.

The use of sponge balls or scrubbing agents is also fairly common. They can provide a better cleaning action than sprays or mixing alone. However, it is important to ensure that the sponge balls will not disintegrate under your cleaning conditions. It's also a good idea to count the balls before putting them in to ensure that they all come out.

Remember to observe the appropriate disposal practices for all used cleaning solutions, which will, in most cases, be considered hazardous waste.

3 Liquid Handling

Contents

PUMPS

Introduction and Terminology	3-2
Pump Sizing and Selection	3-4
Pump Installation and Operation	3-5
Rotary Pump Troubleshooting Guide	3-6
Major Pump Types	3-7
Diaphragm Pump Troubleshooting Guide	3-8
Peristaltic Pump Tubing Sizing and Flowrate Guides	3-13

PIPES, FITTINGS AND VALVES

Pipe, Drawn Tube and Fittings	3-14
Flanges and Gaskets	3-19
Valves	3-20
Hose and Hose Fittings	3-22

FRICTION AND VISCOSITY EFFECTS

Liquid Velocity and Pressure Effects	3-23
Viscosity Effects	3-25

SEALS

Standard O-Ring Size Chart	3-27
Rubber Stopper Sizes	3-28
Ground Glass Joint Sizes	3-28

STORAGE

Capacity of Liquid Storage Tanks	3-29
Storage Drum and Shuttle Data	3-30

Pumps – Introduction and Terminology

Fluid pumping equipment for the CPI continues to evolve, with new developments in design and materials of construction appearing every year. Of particular interest are the expanded use of non-corroding thermoplastics and PTFE or PFA lined pumps, as well as magnetically-coupled seal-less drives which virtually eliminate process fluid leaks. Advances in impeller design offer high efficiency and even the option to retrofit existing equipment with custom-designed impellers to improve performance. It is always worth consulting your pump vendor for an update on current technology.

Pumps fall into two major categories, positive displacement and dynamic. The diagram at the bottom of the page classifies the most important pump types. More detail on the major types is given on the following pages. **Positive displacement** pumps move a fixed volume of fluid for each rotation of a rotor or each stroke of a piston. These pumps can build up extremely high pressures at low operating speeds and therefore should never be run dead-ended (outlet fully closed). Close tolerances prohibit running dry or severe damage could result (peristaltic, bellows, and diaphragm pumps are exceptions). Positive displacement pumps are much better for viscous fluids and most designs are self-priming. Many can also pump backwards by reversing motor direction.

Dynamic style pumps, of which centrifugal pumps are the most important, work by imparting kinetic energy to the liquid, for example by means of a high-speed rotating impeller or blade that generates centrifugal velocity in the liquid. The flowrate is not directly proportional to motor speed (flow decreases as backpressure increases) and because the impeller spins freely in the liquid, at low speeds these pumps will generate no delivery pressure and pump no liquid at all. Viscous fluids have a much more significant impact on the capacity of dynamic pumps than on positive displacement types. However, dynamic pumps in general can move much larger volumes of liquid, albeit at lower pressure, than equivalent-sized positive displacement pumps.

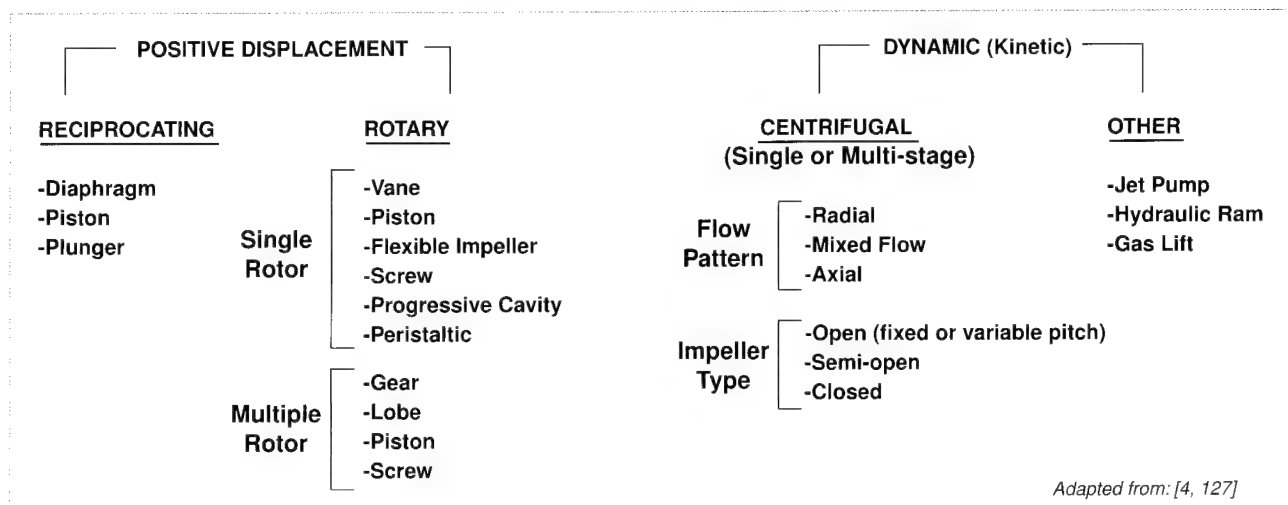
Of principle interest to the CPI are centrifugal pumps and the various rotary styles. Before embarking on a more detailed description and comparison of these types, a review of some basic principles and pump terminology is in order.

Head refers to the pressure experienced at the bottom of a column of liquid of given height. It is typically expressed in feet of water. Because identical columns of liquids with different densities will exert different pressures, a correction for specific gravity must be included when converting from feet of head to other pressure measurements. For example:

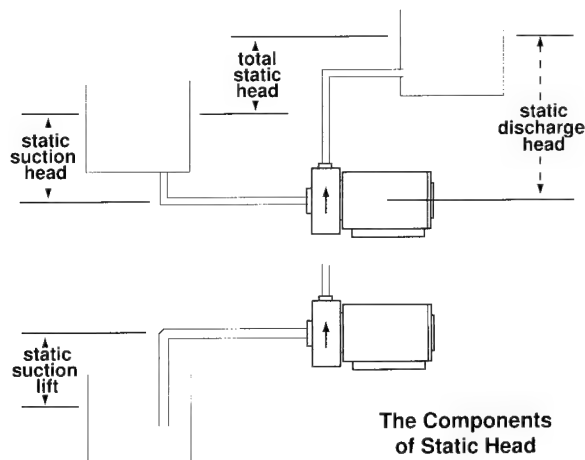
$$\text{Head (feet H}_2\text{O)} = \frac{\text{Pressure (psi)} \times 2.3}{\text{Specific Gravity}}$$

The head against which a pump must work to deliver its product is called the **total dynamic head (TDH)** and it consists of the sum of a number of individual components. These include the total pressures or resistances on the pump discharge

Classification of Major Pump Types



side as well as the pressures or resistances on the pump suction side. The diagram at left shows some of these components. **Static discharge head** represents the vertical height from the pump to its point of free discharge while **static suction head** or **static suction lift** refer to the vertical height from the pump to the free level of the liquid source, either above or below the pump, respectively. The sum of these make up the **total static head**.

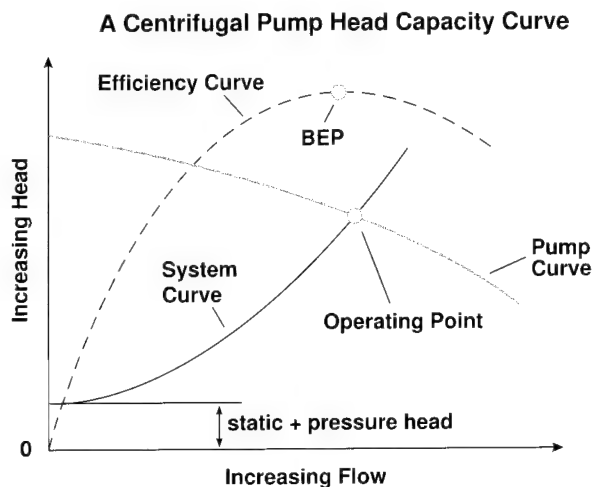


A number of dynamic resistances to fluid delivery must also be considered. These include **friction head**, which represents the pressure the pump must generate to overcome liquid friction in the pipes and fittings to develop its specified flowrate, and **velocity head**, which refers to the added pressure needed to accelerate the liquid from zero velocity to its pumping flowrate. Friction head becomes an important consideration in high-flow situations. Velocity head is often ignored in high-head situations. Additionally, **pressure head** must also be taken into account. This encompasses any other static system pressures which will affect pump operation, for example, if the suction-side vessel is under vacuum or if the discharge-side vessel is at a pressure above atmospheric.

An important but frequently misunderstood term is the **net positive suction head** or **NPSH**. NPSH refers to the *difference* between the pressure at the suction side of the pump and the vapor pressure of the liquid being pumped. If the pressure in the vicinity of the impeller becomes so low that it approaches the liquid's vapor pressure, it can cause the liquid to boil and create the phenomenon known as **cavitation**. This noisy, rapid formation and collapse of minute vapor bubbles can severely reduce pump efficiency, drastically accelerate corrosion in the pump head and lead to mechanical failure. If sufficient NPSH exists, cavitation will not occur. Pump manufacturers often report the NPSH required for proper operation of the pump. Ensuring that sufficient NPSH is available may entail decreasing delivery rate, reducing pump speed, increasing static head, increasing supply-side pressure, operating at a lower temperature or changing the impeller. These details should be discussed with your pump supplier.

The performance of a pump can be evaluated by examining its **head/capacity curve**, which is supplied by the manufacturer. Curves are characteristic of a given pump at a given operating speed. In the figure below the pump curve shows that as head increases, flow decreases, as would be expected. At the zero-flow state (shut-off), the head generated is characteristic of the particular pump and the efficiency drops to zero. Efficiency here refers to the fraction of the energy driving the pump that is actually converted to hydraulic energy to move the fluid. The pump is designed to operate most efficiently at its **Best Efficiency Point (BEP)**. For most centrifugal pumps, the BEP occurs at about 80-85% of the shut-off head value. It is also a function of viscosity and operating temperature. Operating the pump at conditions significantly different from the BEP wastes energy, can increase vibration and wear, and may shorten pump life. That is why regulating flow by throttling the discharge, though workable, is inefficient. Better to reduce motor speed if possible which results in shifting the entire efficiency curve to the left. Regulating AC motor speed requires the use of a **variable frequency drive** (see page 5-13). Another excellent option is to replace the impeller with one designed to match the pump BEP to your normal process flow conditions.

The **system curve**, also shown in the figure, reflects the various heads the pump will encounter in service. It includes static head and pressure heads, which remain constant, as well as losses due to friction in the piping, valves and fittings, which increase with increasing flow. The operating point of the pump is necessarily at an intersection of these two curves. For properly sized pumps this will also be close to the BEP.



Pump Sizing and Selection

Pump selection is based on process flow and pressure requirements as well as the nature of the fluid to be pumped and other system factors. To ensure proper sizing and selection of a pump, work closely with your pump vendor and provide as much information as possible about the application. This will include system curve information, such as static head values and friction losses, along with flow and pressure requirements. Provide any pertinent property data on the fluid or fluids to be pumped, including density, viscosity, flammability, corrosiveness, pH, vapor pressure at the pumping temperature (especially if pumping hot) and the nature and amount of any suspended solids. The expected operating temperature range, the duty cycle (continuous or intermittent), the available power source, as well as information on the environment (indoors, outdoors, hazardous location, etc.) will also play a significant role in the selection. Finally, list any other special needs, such as low shear, self-priming, dry running, operation at or near shut-off, or weight and space limitations. It should be remembered that the initial price of the pump is only a fraction of the total life-cycle operating costs, which include the costs of energy, maintenance, labor, training, and parts replacement. Thus it is well worth the effort to obtain an efficient, properly sized pump for the purpose.

Pump motor size is a key consideration. The chart at the bottom of the page shows the approximate horsepower requirements for small centrifugal pumps as a function of total dynamic head. This can be used as a rough guide in pump sizing, but the performance curves provided by the pump manufacturer are much more accurate since they are based on actual field testing. The horsepower equation given can be used for predicting energy needs for flows and pressures not plotted, and for liquids other than water.

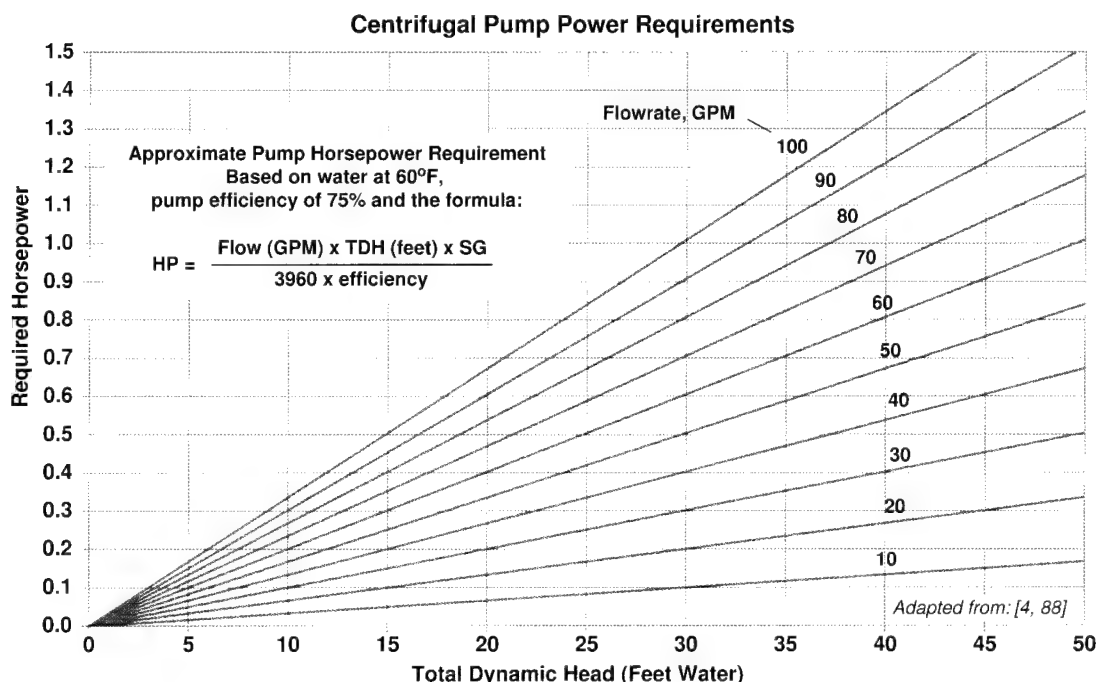
Affinity laws for centrifugal pumps (also called similarity laws) relate pump performance to variables such as motor speed, impeller diameter and power input. They are useful in sizing a pump, and also to predict the effects of a change in operating conditions on an existing pump. The laws are summarized below. Any units may be used as long as they are used consistently throughout. Subscript 1 denotes starting conditions; subscript 2 denotes the new or proposed conditions. Q=flowrate, N=speed (RPM), D=impeller diameter, H=total head pressure, P=input horsepower. Sources [4, 39].

For Constant Impeller Diameter

$$\frac{Q_1}{Q_2} = \frac{N_1}{N_2} \quad \frac{H_1}{H_2} = \left(\frac{N_1}{N_2}\right)^2 \quad \frac{P_1}{P_2} = \left(\frac{N_1}{N_2}\right)^3$$

For Constant Motor Speed

$$\frac{Q_1}{Q_2} = \frac{D_1}{D_2} \quad \frac{H_1}{H_2} = \left(\frac{D_1}{D_2}\right)^2 \quad \frac{P_1}{P_2} = \left(\frac{D_1}{D_2}\right)^3$$



Pump Installation and Operation

Installation – Safe pump operation begins with proper installation, including a stable, properly sized baseplate and mounting, which will prevent vibration and premature failure. Refer to the figure of a typical pump installation below. Piping connections should be well-supported and stress-free to prevent damage and leaks. Never force piping to make a connection. Use bolts of the proper size and materials, be aware of corroded or loose bolts or fasteners and ensure that no fasteners are missing.

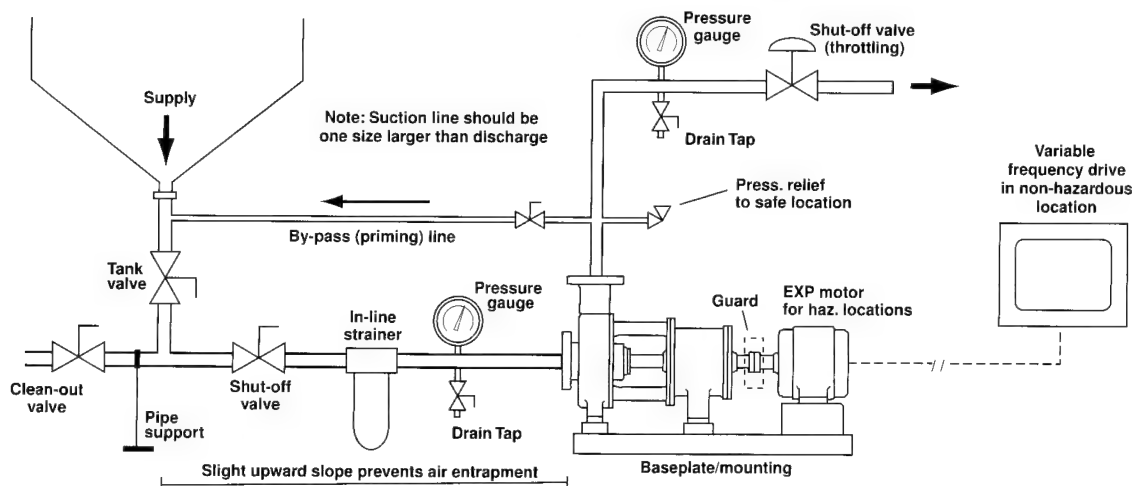
Pipe size is critical, especially at the suction side, to minimize friction losses and maximize NPSH (see page 3-3). The suction side piping should be at least one nominal size larger than the discharge, and it should be as short and straight as possible to prevent poor suction and cavitation (suction-side flow velocities on the order of 5-8 ft/sec are recommended). Elbows or tees located close to the suction side can result in uneven flow distribution, imbalanced operation and dangerous vibration. Keeping air out of the pump is also important for proper operation and long life (only 10% free air in a liquid stream can reduce centrifugal pump capacity by 40%). This can be accomplished by minimizing static suction head and installing suction piping with a slight uniform upward slope toward the pump. No high points or fittings should be present that might allow the accumulation of air pockets.

Service valves should be installed in such a way that the pump can easily be isolated or removed for maintenance if necessary. Suction and discharge pressure gauges should be installed to help ensure proper operation, and an easily maintained in-line strainer should be included upstream of the pump. Ensure that thermal, low flow and overpressure protection systems are properly installed. Relief valves must be vented to a safe location.

Check pump and motor rating plates to ensure that the nominal motor power does not exceed the pump's allowable maximum capacity. For hazardous locations involving the use of flammable liquids or vapors, ensure that the motor is properly rated as to class, group, division and temperature according to NFPA/NEC codes (for more on explosion-proof equipment and definitions of hazardous locations, see page 5-8). Check ease of pump operation by hand if possible and the direction of rotation with the coupling disconnected. Connect the coupling, replace the guard and check for vibration-free operation before introducing process fluid.

Safe Operation – Never operate a pump without the coupling guard (if applicable) in place. Before starting the motor, open the suction valve to flood the pump and open the discharge or priming/recirculation valve. To stop operation, reverse the process, allowing the pump to slow down smoothly. Never operate the pump dry, unless it has been specifically indicated by the manufacturer that this is acceptable. Pumps should not be operated dead-ended, especially positive displacement pumps, although it may occasionally be necessary to operate centrifugal pumps in this fashion for the minimum possible time. Do not throttle the pump with the suction valve, as this can cause cavitation leading to increased

Typical Pump Installation



wear and damage to the pump. Centrifugal pumps may be throttled with the discharge valve, but pumps do have minimum flow limits and operate best near their BEP. In some cases, throttling with the discharge valve can cause excessive temperature rise, high current draw and increased vibration and wear. As a general rule, do not run a pump at less than 50% of its BEP flow for more than 15 minutes. If any unusual symptoms are observed such as noise, vibration, reduced pressure or flow, or overheating, the pump should be stopped immediately until the cause is determined and corrected. The troubleshooting chart below can offer some guidance.

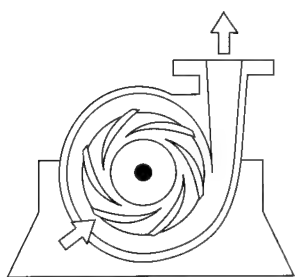
Maintenance – Always lock out power! When working with pumps, always wear appropriate eye protection and insulated or chemically resistant gloves, as appropriate, and observe good chemical hygiene practices. Know the contents of the system. Process liquids in the pump can spray out and cause injury or possibly ignite. Be aware that pump surfaces, bearings and other parts may be extremely hot. Ensure that the pump is isolated from the system and that pressure is relieved prior to disassembly or disconnecting piping. Observe any specific cautions in the pump O&M Manual. To prevent dangerous failures, always schedule preventive maintenance and replacement of wearable parts, elastomer components, gears, motor brushes, etc. according to manufacturer's recommendations.

Rotary Pump Troubleshooting Guide

Symptom →	No liquid delivered	Low flow	Insufficient pressure	Intermittent flow	Bearings hot or failing	Frequent seal failure	Excess vibration / noise	High current draw	Premature wear of internal parts
Possible Cause ↓									
Suction or discharge lines clogged or valves closed	x	x	x				x		
Pump not primed or prime lost	x	x	x	x					
Suction line excessively long or convoluted	x	x	x	x					
Insufficient NPSH (suction head too low)	x	x	x	x		x	x		x
Excessive air in liquid		x	x	x			x		
Air leak or vapor pockets in suction line or pump				x					
Motor speed too low	x	x	x						
Incorrect direction of rotation	x	x						x	
Impeller broken or clogged with foreign matter		x	x		x		x		
Impeller sized incorrectly		x	x						
Discharge head too high		x							
Pump assembled incorrectly			x		x	x	x		x
Shaft/motor misalignment					x	x	x		
Coupling out of balance					x	x	x		
Inadequate/improper lubrication					x				
Suction pressure too high					x	x			
Impeller not balanced					x	x	x		
Incorrect seals or seal installation						x			
Pump is run dry						x			
Pump being run outside design range					x	x	x	x	
Pump running under low discharge load								x	
Baseplate / mounting improperly installed					x	x	x		
Pump operating near system resonant frequency							x		
Worn bearings					x	x	x		
Piping not properly anchored					x	x	x		
Liquid specific gravity too high					x	x		x	
Liquid viscosity too high					x	x		x	
Binding / galling of rotating parts								x	
Chemical incompatibility									x
Abrasives / solids in liquid									x

Adapted from: [111]

Major Pump Types

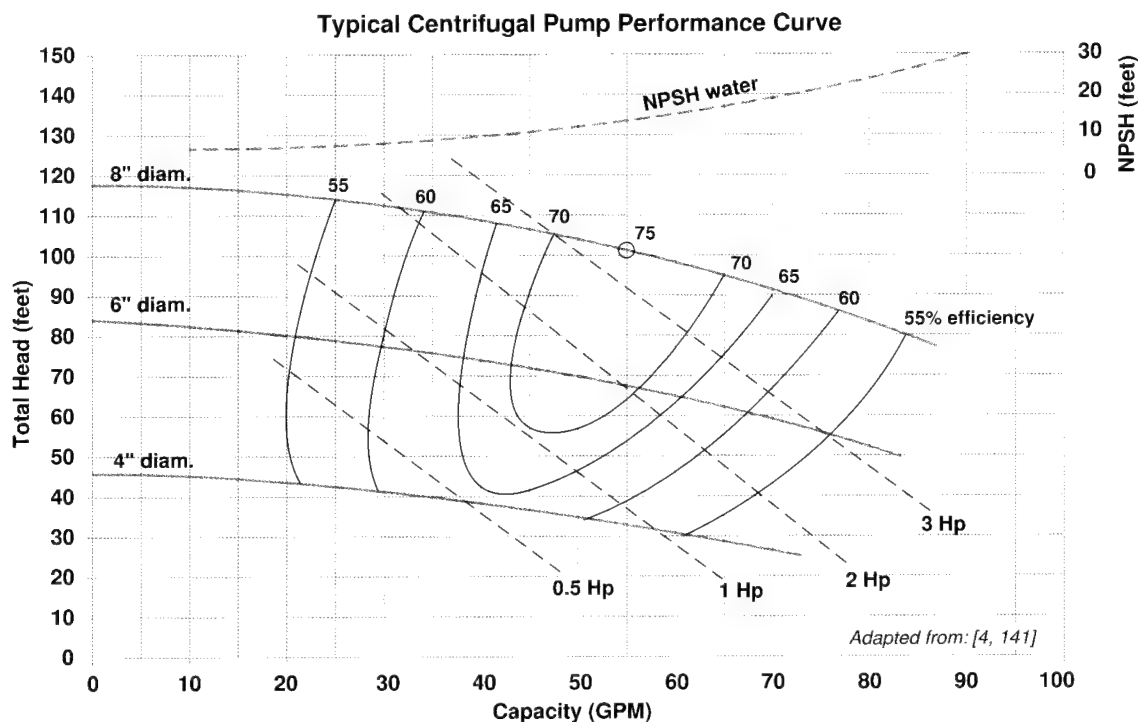


Centrifugal Pumps are by far the most common in the CPI because of their relative simplicity of design, high efficiency, wide flow range and ease of operation. The group covers a broad range of types and a variety of flow/pressure combinations, depending on the design and configuration of the impeller (see figure below). They are generally designed to operate at fixed speed (usually 1725 or 3450 rpm) but flow can be controlled by throttling the discharge, or better, by using a variable frequency drive to regulate speed. Impellers can be reversed to reduce particle attrition when pumping crystal slurries.

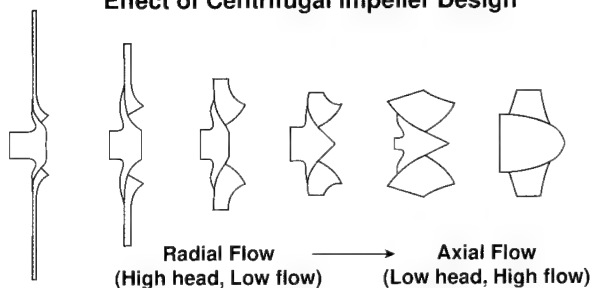
Advantages: Inexpensive, high efficiency, non-pulsatile flow, wide flow and pressure range, some models can be run dry for short periods.

Disadvantages: High speed impellers, thus high shear, not self-priming without flooded suction, priming chamber or recirculation line. Cannot be used with very viscous fluids. Not useful for accurate metering.

Performance Curves – The chart below is an example of a typical manufacturer's performance curve for a centrifugal pump operating at constant speed, showing the efficiencies for various models at various head-flow (H/Q) ratios, as well operating ranges for pumps with various-sized motors and the NPSH as a function of flow. Another common type of pump curve shows performance for a fixed size impeller as a function of motor speed.



Effect of Centrifugal Impeller Design

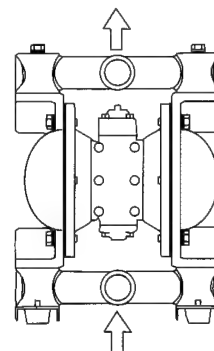


Centrifugal Impeller Styles



Adapted from: [4, 127]

Diaphragm Pumps – These are a type of positive displacement pump widely used in the CPI in cases where pulsed flow is not a concern. Air-driven double diaphragm pumps such as that shown at the right are particularly advantageous in that they are free from the spark potential of electric motors and are therefore ideal for use in hazardous locations. Relatively simple in design, their operation is based on the alternating pressurization and depressurization of two elastomer diaphragms with ball check valves to direct the flow. Capable of very high flows, the total head is limited only by air inlet pressure and membrane material, which must be chosen carefully to optimize mechanical life and chemical compatibility. Major manufacturers include ARO and Yamada. Another important member of this family are diaphragm metering pumps, such as those made by American Lewa. They are accurate and very versatile.



For air-driven models, at an air pressure of 60 psi, a typical 1.5" pump can deliver 60 GPM at 75 feet TDH, and will consume 35 SCFM of air. See the installation and operating tips and troubleshooting guide below. The chart on page 3-26 shows the effect of increased viscosity on pump capacity.

Advantages: Low shear, can be run dry for extended periods, many models are self-priming. Air-driven types are safe for hazardous locations. Relatively inexpensive.

Disadvantages: Pulsatile flow, which without the addition of an external pulse dampener can cause hydraulic shock (water hammer), that can damage piping and other components. Sometimes noisy. Diaphragm rupture is possible.

Installation and Operating Tips – Install an air regulator to control pump speed. Clean, dry, filtered (~5 micron) air should be used. Clean the air filter regularly. Keep air supply pressure at least 15 psi above pump discharge head. However, avoid excessive air pressure, which can cause premature diaphragm failure. In cold weather situations, moisture in the air can freeze and interfere with pump operation. An antifreeze drip line may be installed (consult manufacturer), but keeping air dry (to a dew point of about -40°F) can prevent icing. Keep exhaust muffler clear and replace if it gets wet or clogged with liquid.

Diaphragm Pump Troubleshooting Guide

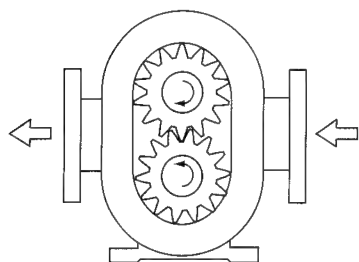
Possible Cause ↓	Symptom →	Not cycling or cycling erratically	Pump cycles slowly	Cycles but no liquid delivered	Cycle with discharge closed	Premature diaphragm failure	Liquid in air exhaust	Air in liquid discharge
Air supply off, or internal air path blocked, or iced up		x						
Suction or discharge lines clogged or valves closed		x		x				
Pump ball check valves stuck or defective		x	x	x	x			
Air filter or air exhaust muffler clogged		x	x					
Air pilot assembly worn or defective		x		x	x			
Air valve seals worn		x	x		x			
Shaft seal o-ring worn		x	x					
Air motor gasket or seals defective		x			x			
Inadequate air supply or undersized air line			x					
Discharge head too high			x					
Supply tank empty				x				
Excessive suction lift			x	x				x
Air leak in liquid suction manifold				x	x			x
Chemical incompatibility						x		
Excessive liquid inlet pressure						x		
Diaphragm used outside design temperature range						x		
Excessive air pressure						x		
Pump assembled incorrectly				x		x		
Failed or ruptured diaphragm							x	x
Excessive air line moisture							x	
Loose shaft nut or center disk							x	x
Liquid specific gravity, viscosity, solids too high			x			x		

Adapted from: [122]

Install inlet and discharge service valves for pump maintenance and inspection. Install a compound (vacuum/pressure) gauge on the suction side and a pressure gauge on the discharge side of each pump to simplify monitoring pump performance and troubleshooting. To minimize pulsations and prevent possible water-hammer damage to piping and other components, a pulsation dampener or expansion tank may be installed in the discharge line. A pressure relief valve should also be installed when pumping liquids with high vapor pressure.

Do not try to prime a dry pump against more than 20 feet of discharge head. It may sometimes be necessary to empty the discharge line to prime the pump if there is a high static discharge head. Installation of a bypass line can also solve this problem.

Avoid excessively high suction pressure, which can cause loud operation and premature diaphragm failure. Keeping inlet pressure less than 15% of discharge pressure is a good rule. When pumping hazardous liquids, flooded suction should be avoided to prevent major leaks in case of diaphragm failure. Place the pump above the supply liquid level in these cases. Remember that capacity will be reduced as suction lift increases as well. A good approximation, useful up to about 20 feet of suction lift, is that for every 3 feet of suction lift, the rated capacity will be diminished by about 5%. A discharge check valve should also be installed to prevent backflow in the event of pump failure.



Gear Pumps – These are another workhorse of the CPI where high pressures (up to 150 psi) and relatively low capacity are required. They are available in two major types, internal gear and external gear. The double external gear is shown in the figure at left. Another popular style is the internal gear pump, in which a single rotating gear meshes with the teeth of a single eccentric set of gear teeth turned inward. This style is more suited to low-pressure, low-flow applications.

All styles of gear pumps work by carrying the liquid in the spaces between the gears and therefore depend on tight meshing and close machining tolerances. Gears can have any number or size teeth, with the design usually being based on the particular

application, flow and pressure requirements. These are positive displacement-type pumps, and the gears are product-lubricated. As with all positive displacement pumps, overpressure relief must be provided. Many models include integral pressure relief systems.

Advantages: Handles very wide range of viscosities, less subject to cavitation than centrifugal pumps. Relatively simple to maintain and rebuild. Nearly pulseless flow.

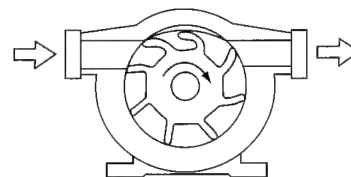
Disadvantages: High shear, close tolerances with possibility of wear or galling, especially if stainless steel construction. Because they are positive displacement, flow cannot be regulated by throttling the discharge, lest excessively high pressures will be generated. Therefore flow control must be accomplished by controlling motor speed with a variable frequency drive (VFD). Requires integral pressure relief valve. Fluid must be free of all abrasives. Cannot be run dry unless gears are made of self-lubricating material such as Ryton.

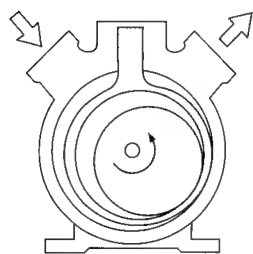
Piston Metering Pumps – These are a popular type of pump used for accurate metering of reagents and other liquids. While they offer the accuracy and reproducibility of diaphragm metering pumps, they have a greater tendency to develop leaks, and therefore are not considered as safe for use with highly toxic or corrosive fluids.

Flexible Impeller Pumps – These are also relatively simple in design, but somewhat limited in terms of pressure and flow (generally less than 40 psi). Depending on the material, impeller lifetime may be limited and is especially susceptible to wear by abrasives in suspension. Impeller wear can also result in product contamination. Impeller is product-lubricated. Positive displacement.

Advantages: Relatively low shear. Non-pulsatile flow.

Disadvantages: Can never be run dry. Flow control must be accomplished at the motor with a variable frequency drive (VFD).





Flexible Liner Pumps – These are a type of positive displacement pump which operate on the principle of an eccentric cam forming a seal with flexible polymer liner. They may also be rather limited in pressure (up to about 40 psi), depending on nature of liner. The liner is fully product wetted and the choice of material for good chemical compatibility is critical. They can handle suspended solids well, but abrasives may wear liner prematurely.

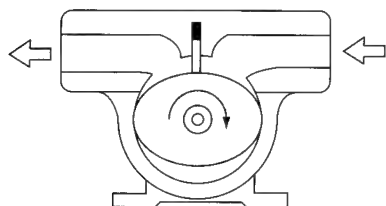
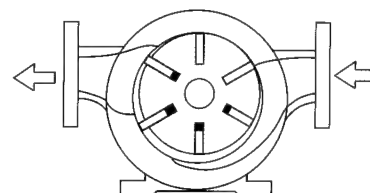
Advantages: Very low shear, handles wide range of viscosities. Self-priming, may be run dry for short periods.

Disadvantages: Pulsatile flow, liner integrity highly dependent on products and materials.

Rotary Vane Pumps – Another widely-used positive displacement style which operates by means of spring-loaded internal vanes on an eccentric cam that slide to maintain wall contact, thereby forming a continuous seal and pockets to carry the liquid. Although vanes adjust to compensate for wear, they must be periodically replaced. These pumps are used for low to medium viscosity liquids (fuel oil, etc.) and are capable of very high flowrates and high pressures. Vanes are product lubricated and will wear prematurely if abrasives are present.

Advantages: High, smooth flow in a relatively small unit, simple design, dependable if maintained. Moderate shear. Can be self-priming if already wetted.

Disadvantages: Limited vane materials available. Preventive maintenance critical to prevent complete vane wear-out and unit failure. Noisy at high speeds. Cannot be run dry.



External Vane Pumps – These are based on a simple design using a single sliding vane which maintains contact with a rotating eccentric cam. The vane must be periodically replaced. Since it is product lubricated, it will wear prematurely if abrasives are present. Should not be run dry. Positive displacement.

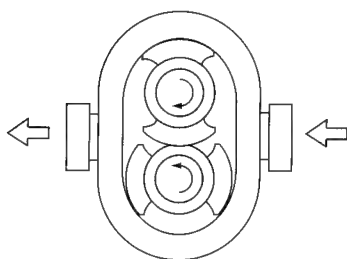
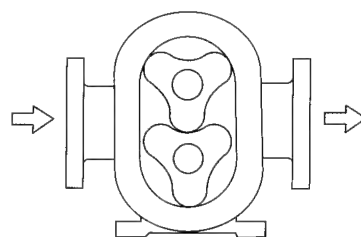
Advantages: High flow rates, non-pulsatile flow, low shear.

Disadvantages: Limited vane materials available. Preventive maintenance is critical to prevent complete vane wear-out and unit failure. Noisy at high speeds.

Rotary Lobe Pump – This is a rather specialized positive displacement design for applications requiring low shear and low operating speeds. Often of all-stainless construction, they are capable of generating high flows at up to 150 psi discharge pressure. They are available in many lobe configurations (3-lobe type is shown here). Non-contacting lobes move liquid in the inter-lobe spaces. Pressure relief required.

Advantages: Very low shear, moderately pulsed flow, low operating speeds and quiet operation.

Disadvantages: Tight tolerances with possibility of wear or galling. Relatively high cost.

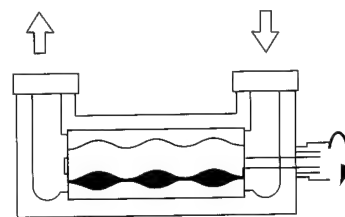


Rotary Piston Pump – This is a specialized pump for low shear, high viscosity and sanitary applications capable of high flows at up to 200 psi discharge pressure. Rotating pistons are designed to be non-contacting and move liquid in the inter-piston spaces. Requires integral pressure relief. Linear performance curves over wide operating range. Positive displacement.

Advantages: Very low shear, moderately pulsed flow, low operating speeds and quiet operation. Handles abrasives and viscous materials well.

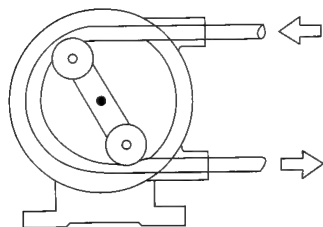
Disadvantages: Tight tolerances with possibility of wear or galling. High cost.

Progressive Cavity Pumps – These are also known as single-screw pumps, this type consists of a double-threaded stator, usually elastomer-lined, and a single threaded rotor which form a series of cavities that progress along the length of the unit as the rotor turns. These are generally very useful for viscous fluids, depending on other properties. They are excellent for abrasive slurries and solids in suspension, especially at reduced speeds where back-slippage is minimized. They should not be run dry. Higher pressures can be attained by increasing unit length (i.e. adding more pump stages). Positive displacement. Moyno is a major manufacturer.



Advantages: High, smooth flow, quiet operation, moderate shear. Self-priming. Rugged and easy to maintain.

Disadvantages: May vibrate at high speeds because of eccentric rotation of rotor. May have starting problems with highly viscous fluids. Cannot be run dry. Fairly large footprint.



Peristaltic Pumps – These are widely used pumps for controlled liquid transfers and chemical metering applications. Also known as “tubing pumps”, they operate by the progressive occlusion of flexible tubing or hose between a set of rollers. They can be used with a wide range of liquids, gases and slurries. Product contacts only the tubing, making it very easy to keep clean and well suited to high purity and corrosive applications. The flow rate is directly proportional to the tubing inner diameter and rotation speed. See the sizing chart for commonly used pump tubing sizes on page 3-13 and the charts of volumetric flowrate vs. rotation speed on page 3-12.

Peristaltic pumps are available in a wide variety of sizes and pumping capacities. Major suppliers of peristaltic pumps include Cole-Parmer, Watson-Marlow and Wanner. Peristaltic pumps are considered positive displacement, but internal leakage and slippage prevents the buildup of excessively high pressures.

Peristaltic pumps handle viscous fluids well, but at reduced capacity. Assume about half the rated capacity for a viscosity of about 500 cP. Flooded suction and short, large-diameter suction lines also help when pumping viscous fluids. For higher pressures, it is wise to choose a smaller-bore and thicker-walled tubing. It is wise to switch to a larger pump model operating at lower rpm and stay with a smaller tubing when higher flows at higher pressures are required. Operating at pressures higher than 30 psi may shorten tubing life.

Advantages: Very low shear. No seals or check valves. Easy to keep clean, and a single pump head can be used for multiple products by simply changing tubing. Handles abrasives well. Self-priming, can be run dry indefinitely. Self-relieving under dead-head conditions. Relatively inexpensive.

Disadvantages: Pulsed flow, unless pulse dampener or special low-pulse pump head used. Tubing must be replaced routinely. Pressure is limited to about 50 psi depending on wall strength, except for larger hose pumps. Chemical compatibility issues may effect tubing life. Relatively low flow rates. Not appropriate for extremely dangerous liquids.

Tubing Selection – The single most important criterion for peristaltic pump tubing selection is chemical compatibility. The table on page 10-10 is a good starting point for assessing the compatibility of some of the more common elastomers with your process fluid. For special situations, more specific information should be obtained from the pump or tubing supplier. For critical applications, the best approach is to test samples of the tubing (usually available from the manufacturer) by immersing them in process fluid as close as possible to the expected process conditions and evaluating the samples for degradation, discoloration, swelling or weight increase. Poor compatibility can drastically shorten tubing life, contaminate the process stream and lead to potentially dangerous failure.

The second most important property of peristaltic pump tubing is long mechanical life. As with so many things, the tubing with the best chemical compatibility is often not the tubing with the best mechanical properties for the repeated flexing that occurs in peristaltic pumps. The chart on the following page can be used to compare the mechanical life of various tubing material types. It should be used as a very rough guide only, since it does not address the tremendous variety of manufacturing methods, proprietary blends and composites, or the availability of braided reinforcement for

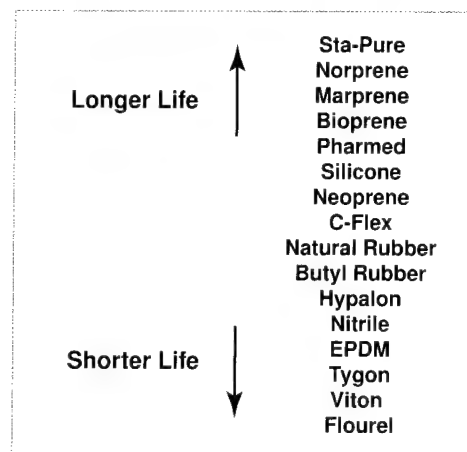
many common types of tube. These factors strongly influence the elasticity and other mechanical properties of tubing.

Note that peristaltic tubing must usually go through a break-in period of an hour or so after which it delivers quite consistent flow for the duration of its life. At the first sign of a drop off of about 25% in throughput, the tubing should be replaced before it fails. Once the lifetime of a given type of tubing is known in a particular application, it is usually very consistent between tubing changes unless process modifications are made which could impact it. Generally speaking, the smaller the bore and the lower the flowrate, the longer the tubing life. Operate at lower speeds when possible.

Never substitute regular grade commercial tubing without consulting the pump manufacturer. Peristaltic tubing is manufactured to tight specifications and tubing which appears identical may not perform satisfactorily. Many pump manufacturers supply special tubing assembly sets to fit their pumps which can be well worth the additional cost to ensure smooth operation without compromising safety.

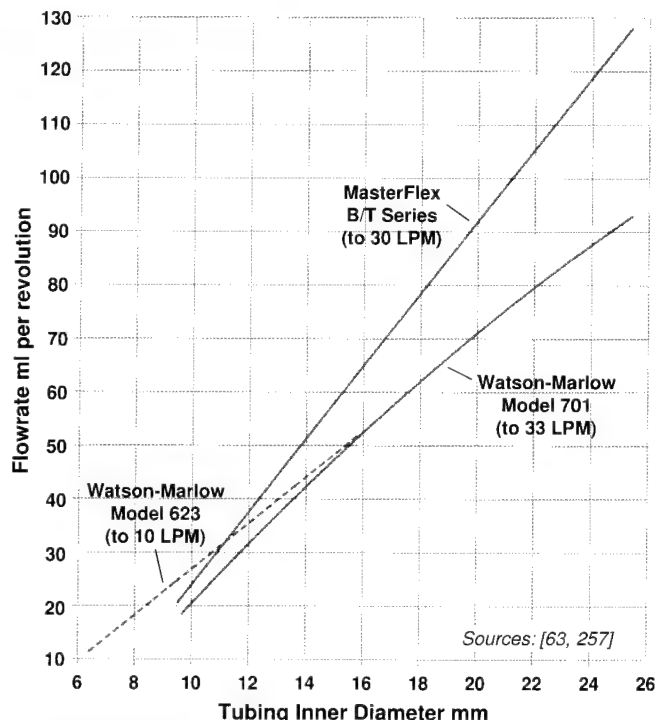
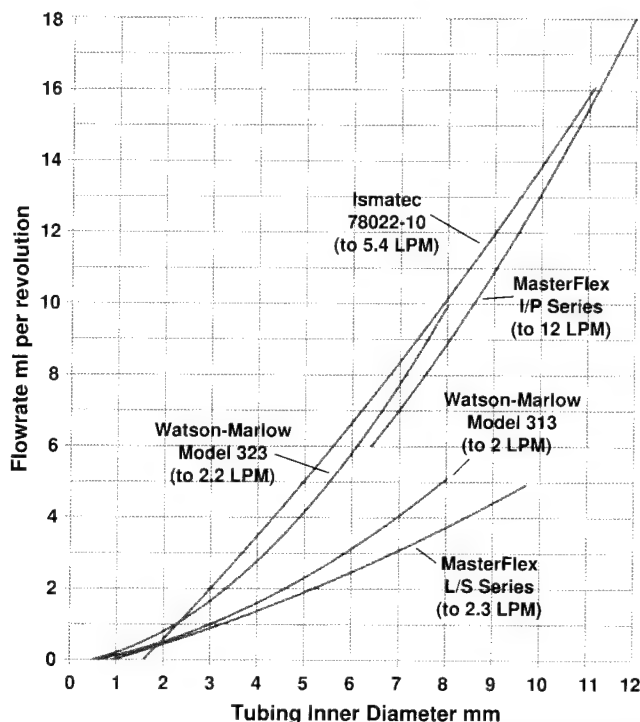
Tubing Size and Flowrate – The flowrate delivered by a peristaltic pump is a function of the tubing ID, the pump head diameter and the pump rotational speed. If the pump speed or speed range is known, it is easy enough to determine what size tubing will deliver the desired flowrate. The charts below correlate pump speed and tubing diameter with flowrate for a number of commonly used pump models. For critical applications, the pump should be calibrated by measuring volume delivered per unit time in an actual test. The chart on the opposite page shows the actual sizes of many common peristaltic tubing types.

Guide to Approximate Relative Peristaltic Tubing Life

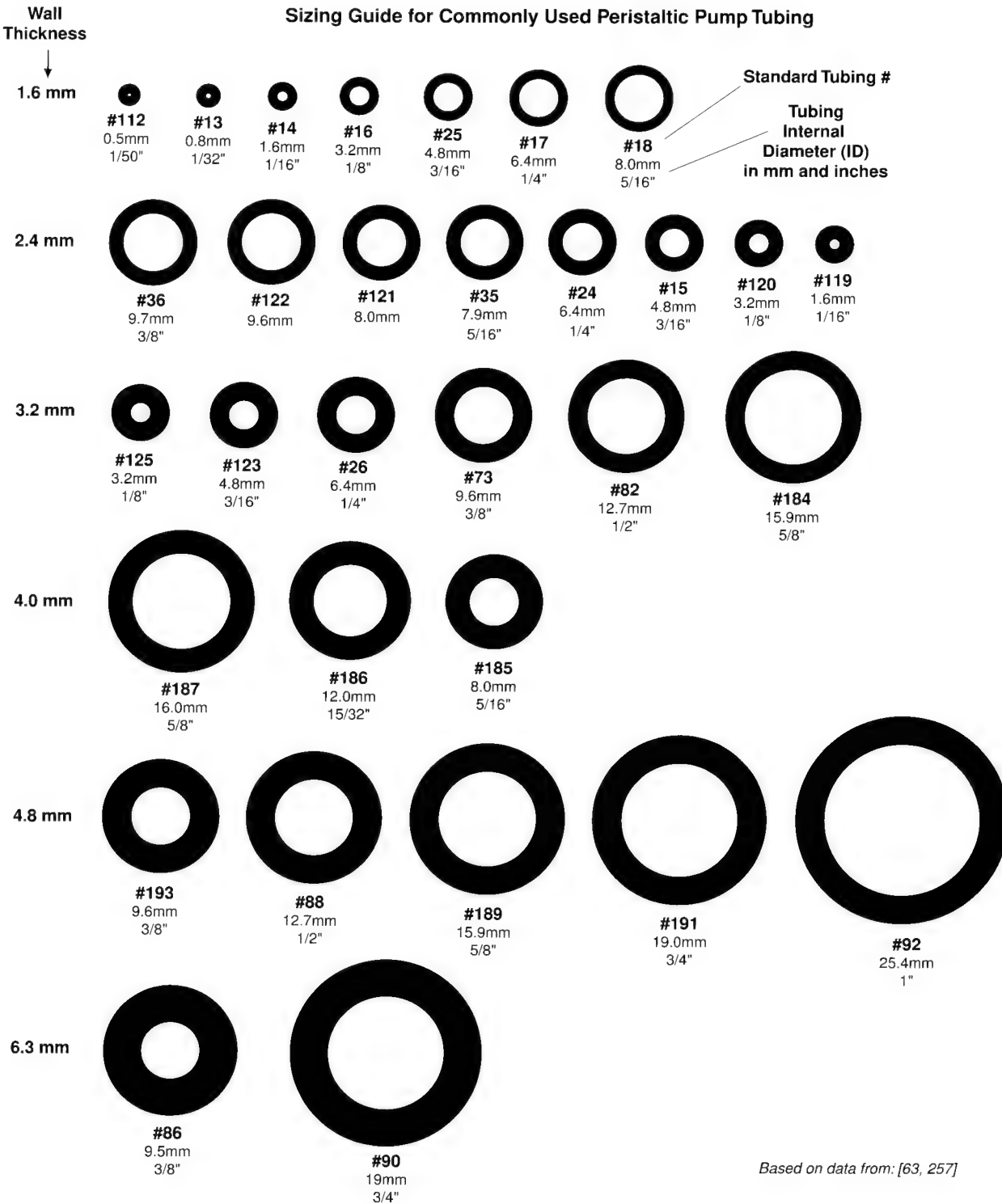


Adapted from: [257]

Approximate Flowrate of Peristaltic Tubing



Sources: [63, 257]



The chart above can be used to visually identify a tube by its dimensions. Sizes that can be used depend entirely on the particular pump-head model. For higher pressure applications, choose the smallest bore size which will achieve the desired flow and the thickest wall available for your model. Consult your pump manufacturer with any questions.

Pipe, Drawn Tube and Fittings

Threaded Pipe – Threaded pipe and fittings is one of the most common types of pipe in use. It is specified by both thread type and size. The most widely used standard in the U.S. is the American Standard Pipe Thread, more commonly called National Pipe Thread, or **NPT**. NPT pipe and fittings are tapered 1.47° to minimize leaks and have a thread angle of 60°. Some suppliers may refer to male and female fittings as **MNPT** and **FNPT**, or by the more confusing shortcuts **MPT** or **FPT**, which mean the same thing. The stated sizes of NPT pipe and fittings, sometimes called **IPS** for Iron Pipe Size, are nominal sizes only and do not reflect the true inner or outer diameter. However, the outer diameter of any given size NPT pipe is consistent across the board regardless of pipe schedule or material. Pipe **schedule** refers to the pipe pressure rating classification system which is based on wall thickness. Schedules 10 through 160 are used industrially; the higher the schedule, the higher the pressure rating. Schedule 40 is the most common for low-pressure applications. The table below lists the actual dimensions of common NPT pipe sizes. The sizing charts at the bottom of the page are helpful in identifying the sizes of NPT pipes or fittings.

Another common standard is the International or **ISO** standard. This is also a tapered thread, but with threads of a different design and angle (55°). Other tapered thread standards often encountered are the **BPT** or **BSPT** (British Standard Pipe Thread) and the **JPT** (Japan Pipe Thread). These various standards cannot be interchanged, although conversion adapters are available for making connections from one set of standards to another.

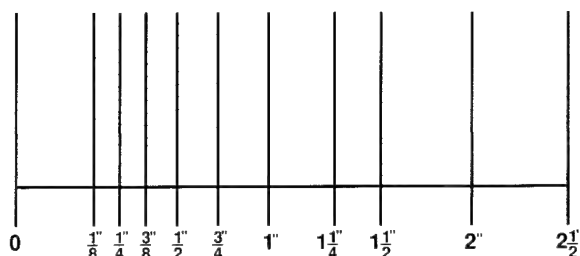
NPT Pipe and Fitting Dimensions

NPT or IPS Nominal Pipe Size	Actual Pipe Outside Diameter		External Surface Area (ft ² /ft pipe)	Pipe Inside Diameter					
				Sched. 40 Steel and Stainless Steel		Sched. 80 Steel and Stainless Steel		Regular Threaded Brass Pipe	
	inches	mm		inches	mm	inches	mm	inches	mm
1/8"	0.405	10.3	0.106	0.269	6.8	0.215	5.5	0.281	7.1
1/4"	0.540	13.7	0.141	0.364	9.2	0.302	7.7	0.376	9.6
3/8"	0.675	17.1	0.178	0.493	12.5	0.423	10.7	0.495	12.6
1/2"	0.840	21.3	0.220	0.622	15.8	0.546	13.9	0.626	15.9
3/4"	1.050	26.7	0.275	0.824	20.9	0.742	18.8	0.822	20.9
1"	1.315	33.4	0.344	1.049	26.6	0.957	24.3	1.063	27.0
1 1/4"	1.660	42.2	0.435	1.380	35.1	1.278	32.5	1.368	34.7
1 1/2"	1.900	48.3	0.497	1.610	40.9	1.500	38.1	1.600	40.6
2"	2.375	60.3	0.622	2.067	52.5	1.939	49.3	2.063	52.4
2 1/2"	2.875	73.	0.753	2.469	62.7	2.323	59.0	2.501	63.5
3"	3.500	88.9	0.916	3.068	77.9	2.900	73.7	3.062	77.8
3 1/2"	4.000	101.6	1.047	3.548	90.1	3.364	85.4	3.500	88.9
4"	4.500	114.3	1.178	4.026	102.3	3.826	97.2	4.000	101.6
5"	5.563	141.3	1.456	5.047	128.2	4.813	122.3	5.063	128.6
6"	6.625	168.3	1.734	6.065	154.1	5.761	146.3	6.125	155.6

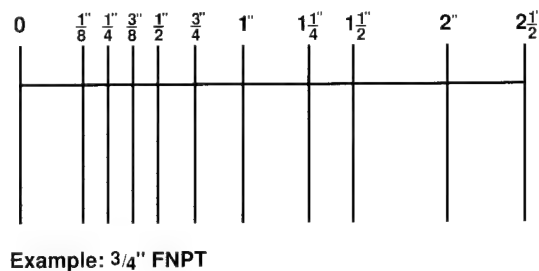
Sources: [88, 172, 194]

Sizing Guide for External NPT Pipe Thread

Example: 1" MNPT



Sizing Guide for Internal NPT Pipe Thread



Adapted from: [172]

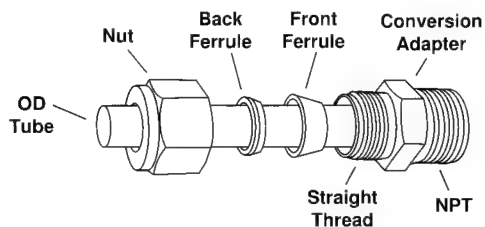
Assembling Threaded Pipe – Threaded pipe is designed to be assembled with a thread sealant and should never be assembled dry, even for a test fit. This is especially true for stainless steel fittings, which have a greater tendency to bind and gall, sometimes making disassembly without thread damage impossible. Teflon tape is a popular and convenient sealant, and teflon-based pipe dopes are also highly recommended. For difficult-to-seal threads, particularly larger-size stainless fittings, the use of both teflon tape and a teflon-based pipe dope is recommended.

Teflon tape should be wound around the male fittings only two to two-and-a-half times, overlapping about half the width for each turn. Using too much tape can compromise the seal. If pipe dope is to be used, apply a small amount over the teflon tape. First hand tighten the joint, and then tighten about one more turn with a wrench or strap wrench. The actual amount to turn and the exact torque value vary widely depending on the condition of the threads, the material and the size of the pipe. Remember that overtightening can create leaks and may permanently damage the threads.

Always ensure that threaded sealing surfaces are clean and free of debris before assembling. Re-using threaded fittings is always risky, unless great care is taken to ensure that the threads are in good condition. For critical applications, it is not recommended.

Straight Thread Fittings – The other common type of threaded fittings use straight, or parallel, threads. These fittings cannot be used to accomplish a fluid-tight seal by themselves. They are designed to be used for mechanical connections only, which include a variety of connections for fluid transport, such as O-ring face seals and compression fittings. The two most widely used standards for straight thread fittings are the American Standard Unified Thread (used in SAE fittings) and the International, or ISO standard. Teflon tape or pipe dope should never be used on straight thread fittings, as the threads themselves are not the sealing point, although they can be lubricated sparingly before assembly to prevent galling and to ease disassembly. SAE fittings are designated by size and thread pitch (threads/inch). ISO fittings are designated by size only, in inches. See the sizing guide at the bottom of the page.

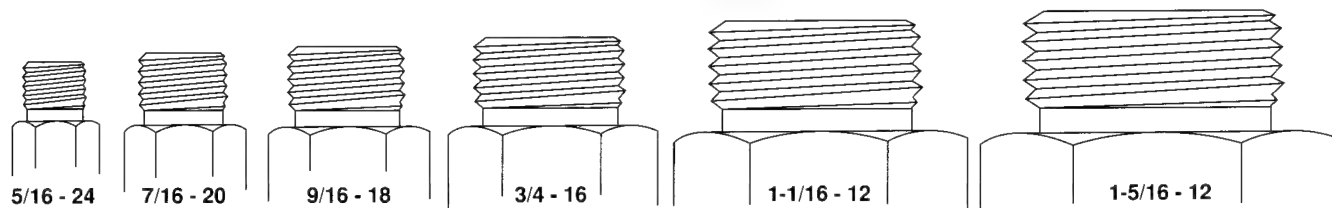
Compression Fittings – These types of fittings are used for making connections to seamless OD tubing (see page 3-16). The most common and reliable are the Parker single-ferrule and the Swagelok double-ferrule types. The pressure rating for metal tube using these systems is very high (on the order of 1000's psi), and they are widely used because of their leak-tightness and convenience. They provide the ability to assemble and disassemble components repeatedly with simple tools without damage.



To ensure proper sealing, it is important that tubing and all fittings are correctly sized and matched and that proper assembly procedure is followed. Do not use ferrules of a softer metal than the tube, for example do not use brass ferrules with stainless steel tubing. Plastic ferrules should be used with plastic tubing. Do not use tubing that is out-of-round or that has surface flats.

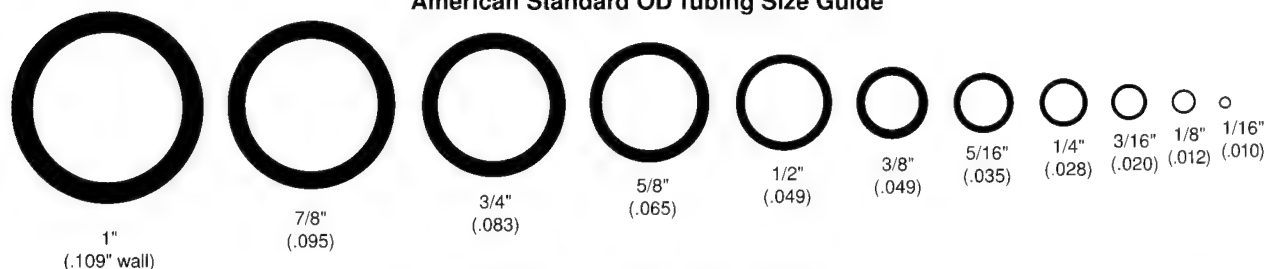
The diagram above shows the proper placement of parts for a typical double-ferrule metal OD tubing assembly. Make sure that all parts are present and that the tube is fully inserted into the body of the fitting until it stops. For new installations using metal ferrules on metal tubing, after snugging by hand the nut should be tightened with a wrench 1 to 1-1/4 turns to fully engage the ferrules. Usually, the tube deforms slightly once the ferrules are fully tightened and seated, and ferrules will then remain in place on the tube even after disassembly. For reassembly, tighten the nut no more than 1/4 turn beyond hand-tight to complete the seal. Do not use teflon tape or pipe dope on ferrules or mating surfaces. Do not overtighten.

American Standard Unified Straight Thread Fittings

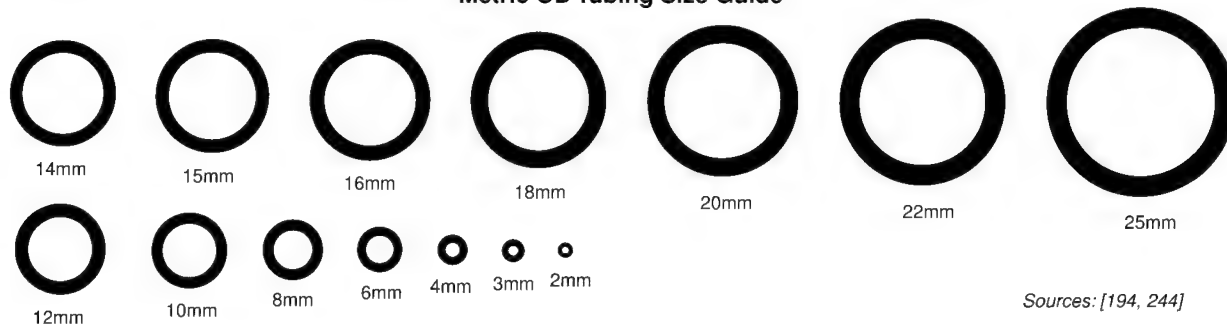


OD Metal Tubing – Outside diameter (OD) tubing is specified in terms of its actual outside diameter for use with soldered or compression-type fittings. Thus, the nominal size of the tube is exactly the outside diameter, regardless of the material. The wall thickness, and therefore the inner diameter, vary depending on the material and on the specific tubing gauge. There are a number of standard wall thicknesses available for most OD tubing, starting at .010" for small size tubing up to .120" or more for 1" or greater tubing. Tubes are rated for pressure duty depending on wall thickness and material. The sizing diagram below shows the most common gauge for each OD tube size, but other gauges are available. A traditional system of metal thickness gauges is sometimes encountered (e.g., .109"=12 Ga, .083"=14 Ga, .065"= 16 Ga, .049"=18Ga, etc.) but it is less ambiguous to state the actual tubing wall thickness in inches.

American Standard OD Tubing Size Guide



Metric OD Tubing Size Guide



Sources: [194, 244]

Copper Pipe – Soft copper pipe is widely used in water, gas and refrigeration service where corrosion is not an issue. It is rated according to its composition, wall thickness and softness. Some common dimensions are listed below.

Soldering Pipe – Copper pipe is often connected by soldering with a low-melting (~190°C) solder. It is sometimes called "sweating" pipe. Always use paste flux, or a flux-cored solder. Clean and roughen surfaces with emery cloth, heat the work first, let the solder flow into the joint and then wipe off excess solder before the joint cools.

Copper Water Tube Dimensions (as per ASTM B88)

Copper Water Tubing Standard Size	Actual Pipe Outside Diameter		External Surface Area (ft ² /ft pipe)	Tube Inside Diameter					
				Type K 400 psi		Type L 250 psi		Type M 250 psi	
	inches	mm		inches	mm	inches	mm	inches	mm
1/4"	0.375	9.5	0.098	0.305	7.7	0.315	8.0	-	-
3/8"	0.500	12.7	0.131	0.402	10.2	0.430	10.9	0.450	11.4
1/2"	0.625	15.9	0.164	0.527	13.4	0.545	13.8	0.569	14.5
5/8"	0.750	19.1	0.196	0.652	16.6	0.666	16.9	-	-
3/4"	0.875	22.2	0.229	0.745	18.9	0.785	19.9	0.811	20.6
1"	1.125	28.6	0.295	0.995	25.3	1.025	26.0	1.055	26.8
1 1/4"	1.375	34.9	0.360	1.245	31.6	1.265	32.1	1.291	32.8
1 1/2"	1.625	41.3	0.425	1.481	37.6	1.505	38.2	1.527	38.8
2"	2.125	54.	0.556	1.959	49.8	1.985	50.4	2.009	51.0
2 1/2"	2.625	66.7	0.687	2.435	61.8	2.465	62.6	2.495	63.4
3"	3.125	79.4	0.818	2.907	73.8	2.945	74.8	2.981	75.7

Sources: [88, 194]

Glass-Lined Steel Pipe – This is widely used in the CPI for transfer of hazardous liquids and process reaction mixtures. It generally consists of Schedule 40 steel pipe in standard lengths which has been flanged on both ends, lined on the inside with a layer of borosilicate glass about 1/16" (1.6mm) thick that has been fused to the metal by high temperature firing. Fittings and adapters of various sizes and configurations are also available. The pipe has excellent chemical resistance to all but highly concentrated acids (especially HF) and hot alkali solutions (see page 10-8) but as with any glass-lined equipment, it cannot be subjected to large sudden temperature changes or mechanical shock (see below). The pressure limit typically ranges from full vacuum to about 150 psi.

Pipe Type	Recommended Differential Temperature Limit
Glass-lined Steel	$\Delta T = 230^{\circ}\text{F}$ (128°C)
Glass-lined Ductile Iron	$\Delta T = 180^{\circ}\text{F}$ (100°C)
Glass-lined Gray Iron	$\Delta T = 100^{\circ}\text{F}$ (56°C)

Source: [198]

Thermal shock occurs when a cold liquid is pumped into a hot pipe, or vice versa. The result can be cracking or flaking of the glass lining or separation of the glass from the base metal. Recommended differential temperature limits for various metal glass-lined pipe are given in the table at left. These numbers should be used as a rough guide only, since geometry and actual operating conditions can affect sensitivity to thermal shock.

Common stock sizes for glassed metal pipe range between 1" to 8" diameter. Fixed length spools (typically 3" to 10 ft in length) and fittings are assembled using special split flanges, usually designed with ANSI Class 150 and 300 dimensions (see page 3-19). Gaskets must be inserted between all glass-lined pipe flanges to prevent damage to the glass surface. Consult the manufacturer for gasket recommendations, but PTFE/Rubber envelope gaskets are usually a safe choice for good mechanical and chemical performance. Bolt dimensions are the same as for ANSI Class flanges, but recommended torque values are generally higher for envelope gaskets. Actual dimensions of common glass-lined pipe sizes and recommended torque ranges are shown below. For more detailed information see [198].

Dimensions of Glass-Lined Pipe

Nominal Size	OD		Approx. ID		Nominal Size	OD		Approx. ID	
	inches	mm	inches	mm		inches	mm	inches	mm
1-1/2"	1.875	47.6	1.50	38	4"	4.50	114	3.900	99.1
2"	2.375	60.3	1.95	50	5"	6.63	168	5.950	151.1
3"	3.500	88.9	2.95	75	6"	8.63	219	7.850	199.4

Adapted from: [198]

Recommended Flange-Bolt Torque Values for Glass-Lined Pipe Connections

Pipe Size	Recommended Torque Value
up to 1-1/2" pipe	1/2" bolts - 20-30 ft-lbs
2", 3" & 4" pipe	5/8" bolts - 40-60 ft-lbs
6" & 8" pipe	3/4" bolts - 70-100 ft-lbs

Adapted from: [198]

Borosilicate Glass Pipe – This is a common choice in the CPI where corrosion resistance is required, but it also offers other advantages such as its transparency, low thermal expansion ($\sim 0.4''$ per 100 ft per 100°C , about $\frac{1}{4}$ of that of steel pipe), relatively high operating temperature (up to 230°C for the glass itself, but joint and shielding materials must be considered), UV transparency and suitability for high purity applications. However, glass pipe is also subject to thermal shock due to sudden temperature change. A differential of 100°C should be considered the safe limit for pipe under 3", 80°C for larger pipe.

Glass pipe is available in a number of configurations, including plain-end, the widely used beaded-end, conical-end, and beaded-armoured, each with specifically designed joints and connectors. The non-glass components, such as seals and insulation, must be selected carefully based on operating conditions. As with any non-conducting material, glass pipe must be electrically grounded to prevent the buildup of dangerous levels of static charge. Consult the manufacturer for details on proper grounding procedures. Glass pipe must never be subjected to bending stresses. Careful alignment and proper pipe support systems are important. Valves and other heavy components should be supported directly. The external surface of the pipe should be protected against scratches or other surface defects which can weaken the pipe.

Rigid Plastic Pipe – PVC and CPVC rigid plastic pipe are widely used because of the many advantages they offer for certain applications—high mechanical strength, low weight, immunity to electrolytic or corrosive attack, good pressure rating, low thermal conductivity, lower friction head loss than steel pipe, and easy on-site assembly by solvent welding. However, it is not suitable for use with aromatic or chlorinated solvents, acetates, esters, ketones and other non-polar organic liquids (see the table starting on page 10-10). Rigid PVC is often referred to as Type 1 PVC. This distinguishes it from soft PVC, such as the kind used to manufacture tygon tubing, which has added plasticizers.

Schedule 40 and 80 PVC and CPVC pipe have the same actual inner and outer diameter as Schedule 40 and 80 steel pipe (see page 3-14). CPVC has a maximum temperature rating of 200°F (93°C), PVC a maximum rating of 140°F (60°C).

Pressure ratings for various PVC pipe classifications and sizes are listed at the bottom of the page along with charts showing temperature de-rating factors. The pressure ratings in the tables are for water at 73°F (23°C). These ratings must be multiplied by the de-rating factors from the chart for use at higher temperatures. The ratings also assume that proper assembly procedures were followed (see below), and proper pipe support systems (roughly every 4-6 ft) are used.

Thermal expansion is also more of an issue for rigid PVC than for metal piping systems. Pipe length will typically increase about 0.5% per 100°F increase in temperature. Piping systems should also be designed to keep fluid velocities

below 5 ft/sec, which will minimize the possibility of damage from hydraulic shock due to quickly-closing valves, etc.

Plastic Pipe Type	ASTM Designation
Sch. 40, 80, 120 PVC	D-1785
SDR (Pressure Rated) PVC	D-2241
Drain, Waste and Vent (DWV) PVC	D-2665
Sch. 40, 80 CPVC	F-441

The standard **markings** found on PVC pipe include: manufacturer's name, material code, nominal size,

schedule, pressure rating (psi) for water at 73°F (23°C), ASTM designation number (see table at left), NSF seal for potable water, and a manufacturing date code. Source [226].

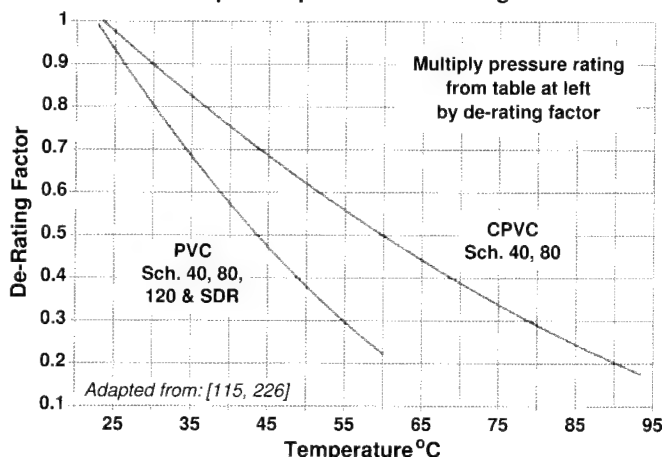
Frictional head loss due to flow through rigid PVC pipe is slightly lower than that for equivalent flow in steel pipe because of the high smoothness of the inner wall. For estimating purposes, assume that losses will be approximately 20% lower than the values shown on page 3-24 for equivalent lengths of Schedule 40 steel pipe and fittings.

Assembly – PVC and CPVC pipe are most commonly assembled by solvent welding using designated PVC solvent cements, although NPT threaded fittings are also available for Schedule 80 CPVC pipe. For solvent welding, cut the pipe with a fine-tooth saw or wheeled cutter (ensure no raised bead is left on pipe end). Chamfer the pipe ends slightly, and remove all grease, dirt and moisture with a clean rag. Apply primer to both the pipe and socket surfaces using a natural bristle brush until the surfaces are well penetrated. Again using a natural bristle brush apply a liberal coating of solvent cement to both surfaces. While both surfaces are still wet, seat the pipe all the way to the bottom of the socket with a 1/4-turn twisting motion. The cementing operation should be accomplished quickly to prevent cement from drying. Joints set within minutes, but follow the cement label instructions for recommended cure and handling times.

Pressure Rating (psi) for Water at 73°F (23°C)

Nominal Pipe Size	Sch. 40 PVC Type I and CPVC	Sch. 80 PVC Type I and CPVC	SDR 41 all sizes 100 psi
1/4"	780	1130	
3/8"	620	920	
1/2"	600	850	
3/4"	480	690	
1"	450	630	
1 1/4"	370	520	
1 1/2"	330	470	
2"	280	400	
2 1/2"	300	420	
3"	260	370	
3 1/2"	240	350	
4"	220	320	
			SDR 26 all sizes 160 psi
			SDR 21 all sizes 200 psi
			SDR 13.5 all sizes 315 psi

Plastic Pipe Temperature De-Rating Factors



Flanges and Gaskets

Flanged connections are a common means of joining pipes and equipment, and are available in a variety of materials and styles. Flange specifications are set by ANSI. Dimensions shown in the table below are standard for all types of steel flanges. The current classification system replaces the traditional "pound" rating system, i.e. a "Class 150" flange is equivalent to a "150-lb" flange. The pressure ranges given below provide a general idea of how the classification system works, but the actual pressure rating depends on the specific material of construction. Diagrams of some of the common flange types are also given below. Raised face (RF) flanges provide better contact and pressure containment than flat face (FF). It is not necessary to use two raised face flanges – an RF against a FF provides the same benefit as two RF, at lower cost. For leak-prone applications, Class 300 flanges are recommended since they allow higher bolt stress.

Gaskets – The asbestos gasket was the standard in the CPI for many years. It offered wide chemical compatibility and fire resistance at a low cost. For health reasons, however, asbestos has been replaced by a number of other materials. PTFE, aramid fiber bound with NBR, SBR or neoprene, fiberglass, laminated graphite and carbon or graphite fiber are just some of the materials now used. Each offers its particular advantages in terms of leak control, temperature and

Dimensions of Common Flange Sizes

Nominal Pipe Size	Class 150 Steel Flanges				Class 300 Steel Flanges			
	Flange OD	Thickness	Bolts	Bolt Size (diam)	Flange OD	Thickness	Bolts	Bolt Size (diam)
1/2"	3 1/2"	7/16"	4	1/2"	3 3/4"	9/16"	4	1/2"
3/4"	3 7/8"	1/2"	4	1/2"	4 5/8"	5/8"	4	5/8"
1"	4 1/4"	9/16"	4	1/2"	4 7/8"	11/16"	4	5/8"
1 1/4"	4 5/8"	5/8"	4	1/2"	5 1/4"	3/4"	4	5/8"
1 1/2"	5"	11/16"	4	1/2"	6 1/8"	13/16"	4	3/4"
2"	6"	3/4"	4	5/8"	6 1/2"	7/8"	8	5/8"
2 1/2"	7"	7/8"	4	5/8"	7 1/2"	1"	8	3/4"
3"	7 1/2"	15/16"	4	5/8"	8 1/4"	1 1/8"	8	3/4"
3 1/2"	8 1/2"	15/16"	8	5/8"	9"	1 3/16"	8	3/4"
4"	9"	15/16"	8	5/8"	10"	1 1/4"	8	3/4"

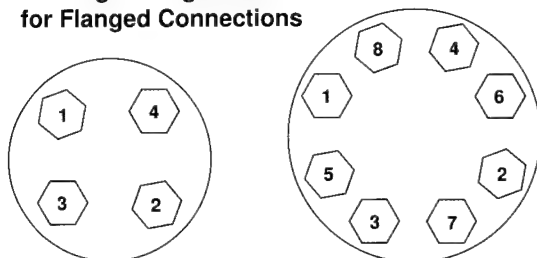
Recommended Torque: 1/2" bolts – 10-15 ft-lbs 5/8" bolts – 20-30 ft-lbs 3/4" bolts – 33-50 ft-lbs

Pressure Ratings of Class 150 and 300 Flanges

Temp. Range °C	Class 150 Steel Flanges
-20 to 100	230 - 290 psi
200	195 - 260 psi
300	175 - 230 psi

Temp. Range °C	Class 300 Steel Flanges
-20 to 100	600 - 750 psi
200	505 - 750 psi
300	455 - 730 psi

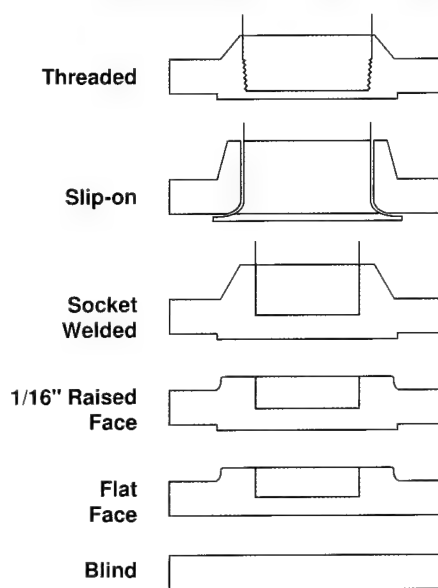
Bolt Tightening Patterns for Flanged Connections



Four Bolt Pattern

Eight Bolt Pattern

Common Types of Pipe Flanges



Sources: [49, 88, 160, 194]

pressure range, and chemical resistance. Gaskets can be cut from sheets by the user, but preformed gaskets assure better performance. Preformed gaskets usually consist of several components of different materials designed to provide enhanced sealing capability. Metal spiral-wound gaskets are one such example. Often referred to as Flexitallic gaskets because of their original manufacturer, they consist of a thin metal strip wound edgewise surrounded by various elastomeric filler materials and offer excellent leak performance in difficult situations. Other preformed gasket types include graphite/corrugated metal, double jacketed and envelope gaskets. For more complete information on gasket selection, contact any reputable gasket vendor.

Assembling Flanges – When assembling, lubricate bolt threads and use flat washers under every nut and bolt head. Ensure that the pipes line up correctly and that the gasket is properly centered. Snug the bolts by hand and then gradually tighten to the indicated torque following the bolting pattern given on the previous page. Completely tightening one or two bolts before the others are snugged down can pinch or otherwise damage the gasket and cause leaks. Rather, tighten all bolts gradually following the crisscross pattern to ensure even compression of the gasket and to avoid otherwise bending or stressing the connection. Also, because of bolt elongation and gasket compression, the bolts should be checked and retightened if necessary once the system has been in operation for a while, and periodically thereafter. Finally, when non-conducting gaskets are used in applications involving flammable liquids, flange-grounding jumper wires must also be installed to provide electrical continuity along the fluid path.

Valves

Valves for fluid control come in many styles. Brief descriptions of some of the more common types are given below. Many of those listed are available with electric or pneumatic actuators for automated operation, particularly ball and butterfly valves in which complete actuation is accomplished with a 90° turn of the handle. Much more detailed information on the construction, application and safety features of various valve types can be found in [160].

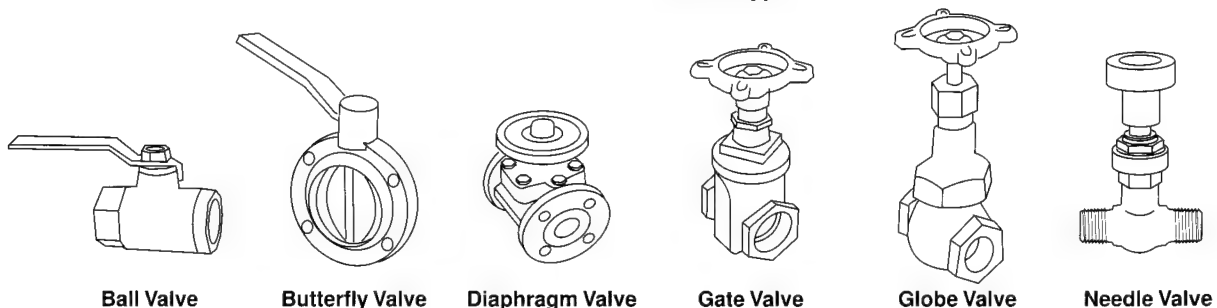
Ball valve – A simple, dependable general service valve. Available as threaded, flanged, socket welded, and in a tremendous variety of body, ball, and seat materials, including all-plastic or PTFE construction, and in single-piece or multi-piece styles to simplify maintenance. Best for on-off service, but can also be used as crude throttling valve. Full-port styles offer very low flow restriction. Actuates with a 90° turn of the handle. Handle position indicates valve status.

Gate valve – Works by incrementally imposing a flat “gate” into fluid stream as handle is turned. Also low friction when fully open. Best for on-off duty, not recommended as throttling valve. Rising-stem style gives a visual indication of valve position, but non-rising stem style may be better when space is limited.

Globe valve – Named for the spherical shape of the valve body, these valves control flow by means of a flat plug or disk located in an internal baffle. The baffle restricts flow and therefore creates backpressure even when the valve is fully open. These valves open and close more quickly by handle action than gate valves, and thus are useful in applications requiring frequent operation.

Butterfly valve – Flow is controlled by means of a round disk that rotates at a 90° angle to the direction of flow. When fully open, the disk is set parallel to the flow. Low backpressure is generated, slightly more than for a gate valve or ball valve, but only about 1/3 of that generated in a fully open globe valve. A less expensive alternative to ball valves,

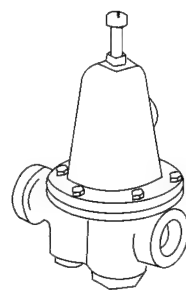
Some Common Valve Types



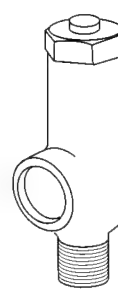
especially in large sizes. They can be used in on-off and throttling service. Actuates with a 90° turn of the handle.

Needle valve – Similar in construction to a globe valve, but with a finely tapered plug or needle designed to provide very precise control over flow of liquids and gases. Usually available only in smaller sizes.

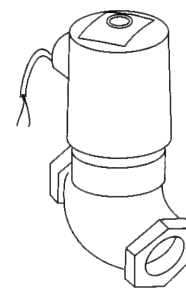
Diaphragm valve – Flow is controlled by an elastomer diaphragm which is squeezed into the fluid stream by handle action, thereby constricting flow. Very precise throttling is possible. They handle slurries and suspended solids well. Durable and low-maintenance. These valves are often specified for sanitary service because their smooth internal design generates low fluid shear and provides few places for contaminants to accumulate.



**Pressure
Regulator**



**Pressure
Relief Valve**



**Solenoid
Valve**

Solenoid valves are simple electrically actuated on-off valves. A valve stem is moved in or out of the fluid stream by the direct action of the solenoid coil. **Normally-open** solenoid valves will close only when the coil is energized by electric current and **normally-closed** valves will open only when the coil is energized. Safe practice often dictates the use of normally closed valves so that in the event of a power outage, no key valves are inadvertently left open, but a comprehensive automated control plan, even for simple systems, should be thought through very carefully.

Relief valves are mechanical devices designed to protect vessels, piping and other components by relieving excess gas, steam or liquid pressure in an overpressure situation. They automatically pop open when pressure exceeds a preset level and remain open until the pressure drops back below that level. No mechanical device is perfect, and often relief valves begin to leak as pressure approaches the preset value, and may continue to leak slightly even after pressure has dropped back down to a safe level. An overpressure of about 20% may be required to fully open the valve. Some relief valves have an adjustable setpoint. Others, such as ASME-coded valves (recommended in critical applications), are factory preset and cannot be adjusted. It is also important that the valve outlet be directed to a safe location.

Check valves – these devices are designed to allow flow of fluid in one direction only. A wide variety of styles exist, including **spring checks**, **ball checks**, **duckbill checks**, **y-checks**, **swing** or **flap checks**, among others. Usually, a minimum “cracking pressure” is required to start forward flow, especially in the case of the spring-loaded check valves. Most offer moderate flow resistance, with swing checks being the lowest, spring-loaded or gravity-lift styles being the highest. Ball checks are recommended for viscous liquids and duckbill checks are most suitable for slurries or suspended solids. Depending on the style, the valve may or may not be suitable for both horizontal or vertical piping.

Pressure regulating valves (pressure regulators) are designed to reduce and regulate pressure. They are available with adjustable or factory preset pressure setpoints. Some models divert excess liquid flow back to the supply via a bypass loop, others simply throttle flow. Selection must be made with consideration for the type of pumping system, the nature of the fluid, material compatibility, the effect of process temperature on the pressure setpoint, etc. To properly size the valve, one must know the pipe size, maximum inlet pressure, desired outlet pressure, and required flow rate. With this information, the valve manufacturer can help select the appropriate valve. As a general rule, the valve should be sized so that it normally operates at about 65% of its rated maximum flow.

Valve packings – Graphite, polymeric composites, braided carbon fibers and other materials are now used in place of the traditional asbestos valve packings. These modern alternatives offer longer life, improved pressure ratings, improved leak performance and higher service temperatures. Because of the wide variety of available styles, you should discuss any special needs or material compatibility issues with your valve manufacturer or supplier.

A number of other specialty valves such as plug valves, gas cocks, hydraulic control valves, vacuum-breaking valves, toggle valves, mixing valves, and directional valves are available for special applications. The McMaster-Carr Supply Company product catalogue [172], which includes diagrams of many valve types, dimensions, and application information, is an excellent resource for selecting the proper valve or valve accessory for your application.

Hose and Hose Fittings

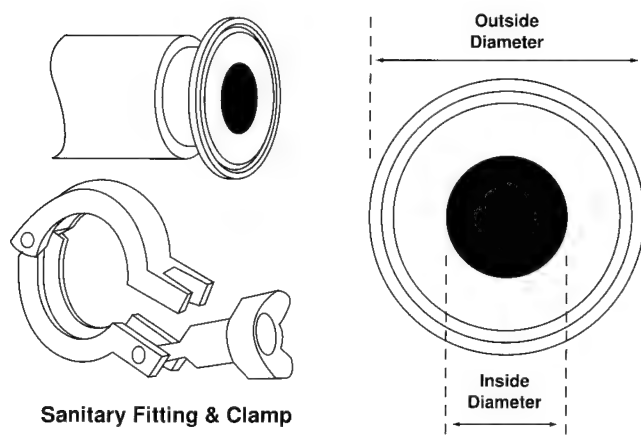
Industrial hose is available in a tremendous variety of styles, sizes and materials to cover wide pressure, temperature and chemical service ranges. Plain hose, lined hose, inner-braided hose, and stainless overbraided hose are just a few of the styles in common use. Pre-made hose assemblies are manufactured with flanged, threaded, compression, bevel seat, sanitary, camlock and many other types of termination to meet any fluid transfer need. Therefore, the selection of the proper hose is not trivial and careful consideration should be given to expected operating conditions, chemical compatibility, pressure, temperature, hose movement, the type of installation and the frequency with which the hose connections will be made and unmade. Any reputable hose supplier will be able to assist in your hose selection.

Installation and Use – Proper installation is as important as selecting the proper hose. Before use, always inspect hose for damage such as cover abrasions, cuts, kinks or crushing of the hose, all of which can reduce the life and pressure rating. Never use damaged hose. If hose will need to flex, limit the movement to a single plane to minimize twisting or torquing. Twisting or torquing should also be prevented during installation. Floating flanges or swivel fittings can accommodate twisting if necessary. All bends should be smooth with no sharp turns, which can cause kinking and damage the hose. If sharp turns are necessary, the use of properly designed elbows is encouraged. Remember that excessive or repeated bending can induce stress fatigue. No axial movement (stretching or compressing along its length) should be allowed. External abrasion and wear due to vibration and contact with other surfaces should also be avoided. Finally, hose connections and terminations should be kept square and free from bending, which can compromise the seal and cause leaks and eventual failure of the joint. When cam-lock fittings are used, always make sure that the safety rings are secured to prevent the connection from accidentally opening during use.

Grounding – When non-conducting hose is to be used to transfer flammable solvents, ensure that the hose is properly grounded to prevent the buildup of dangerous static charges on the hose. This may involve winding the outside of the hose with an unshielded grounded wire, or in some instances, installing a grounding wire inside the hose. Static charge buildup is a particular concern when very non-polar solvents such as hexane and heptane are pumped at high speeds.

Hose Fittings – Any industrial supply catalogue will list the various types of fittings and materials available. Each style has its advantages, but the user should give careful thought to the expected operating temperatures and pressures, the selection of gasket material and cleanability of the fitting. Pre-made hose assemblies are almost always the safest choice.

Sanitary Hose Fittings – Sanitary hose fittings are one type of connection widely used in the food, dairy and pharmaceutical industries wherever connections must be broken frequently and where cleanliness and smooth internal fluid path are important. Specifications are set forth in the 3A Sanitary Standard which is a voluntary standard set by the IAFIS. Various types of sanitary connections are in use including the bevel-seat and I-Line systems, but the Tri-clamp is the most common. Gaskets are available in a wide variety of elastomers. Sanitary fittings are not recommended for high pressure, high temperature or corrosive chemical service. The figure below shows a typical Tri-clamp sanitary connection and the table lists dimensions for the most common sizes. Note that the 1/4", 3/8", 1/2" and 3/4" "mini" fittings have the same outside diameter, as do the 1" and 1 1/2" sizes.



Dimensions of Tri-Clamp Sanitary Fittings

Nominal size	Outside Diameter inches	Outside Diameter mm	Inside Diameter inches	Inside Diameter mm
1/4 "	0.984	25.0	0.187	4.7
3/8"	0.984	25.0	0.227	5.8
1/2"	0.984	25.0	0.375	9.5
3/4"	0.984	25.0	0.625	15.9
1"	1.984	50.4	0.870	22.1
1-1/2"	1.984	50.4	1.360	34.5
2"	2.516	63.9	1.870	47.5
2-1/2"	3.047	77.4	2.370	60.2
3"	3.579	90.9	2.870	72.9

Adapted from: [220]

Liquid Velocity and Pressure Effects

Chart for Estimating Velocity of Liquid in Pipes

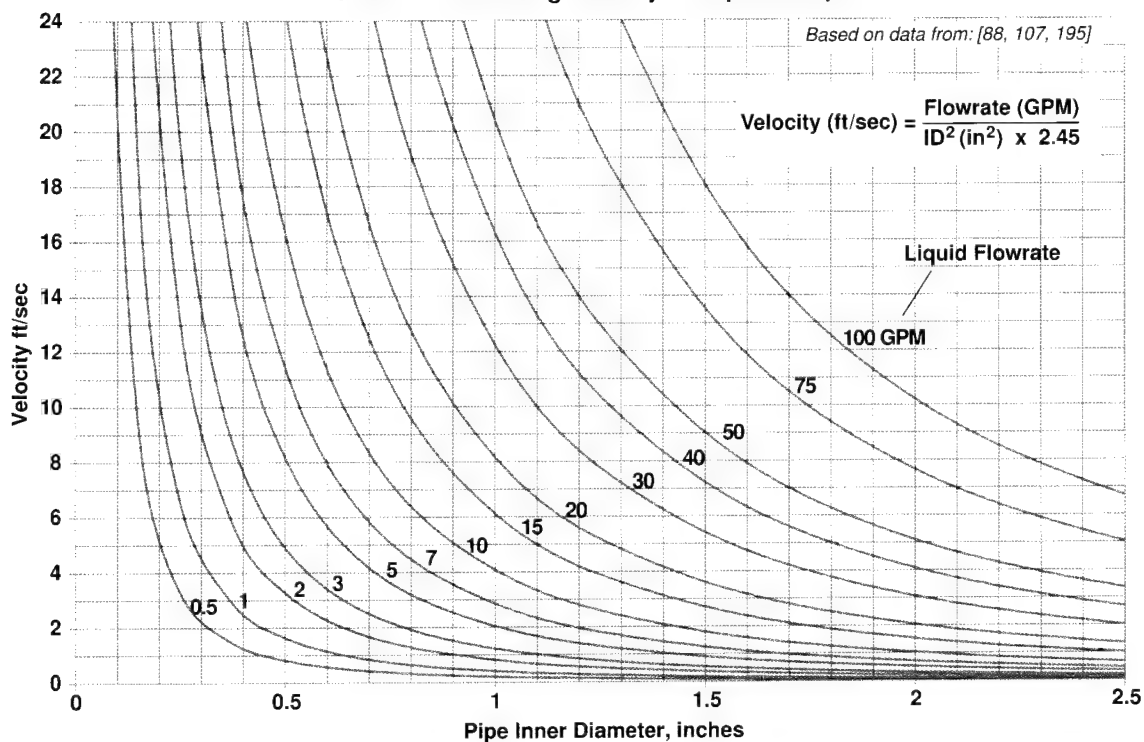


Chart for Estimating the Flowrate of Liquids Through an Orifice

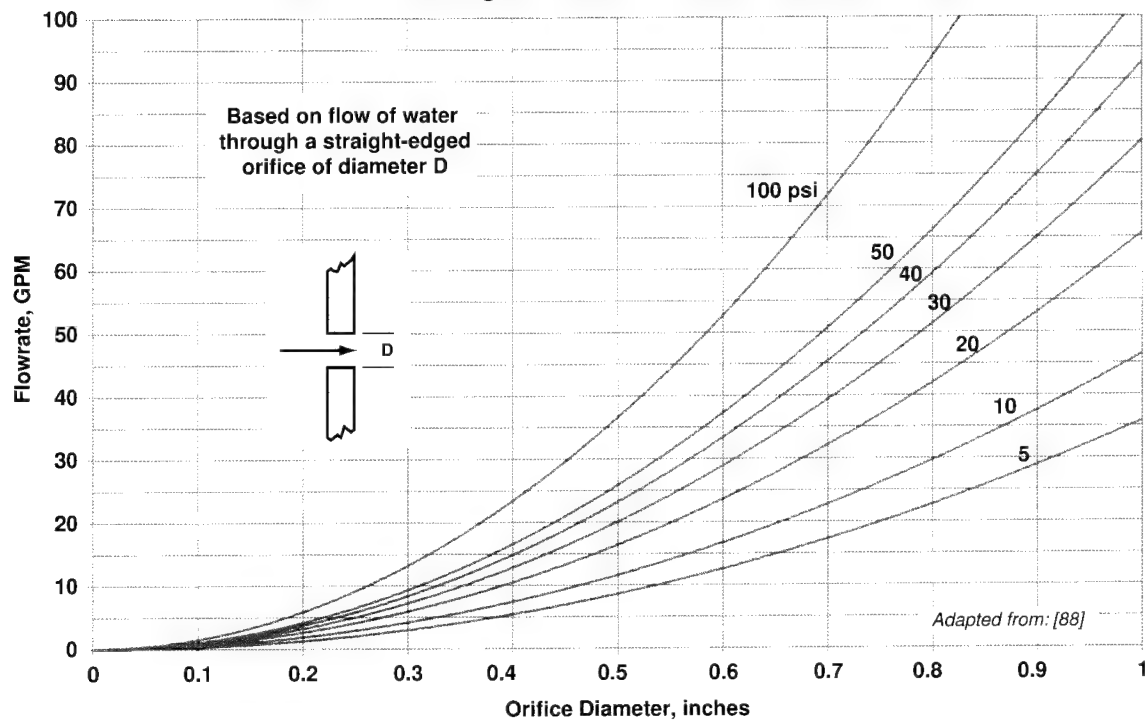
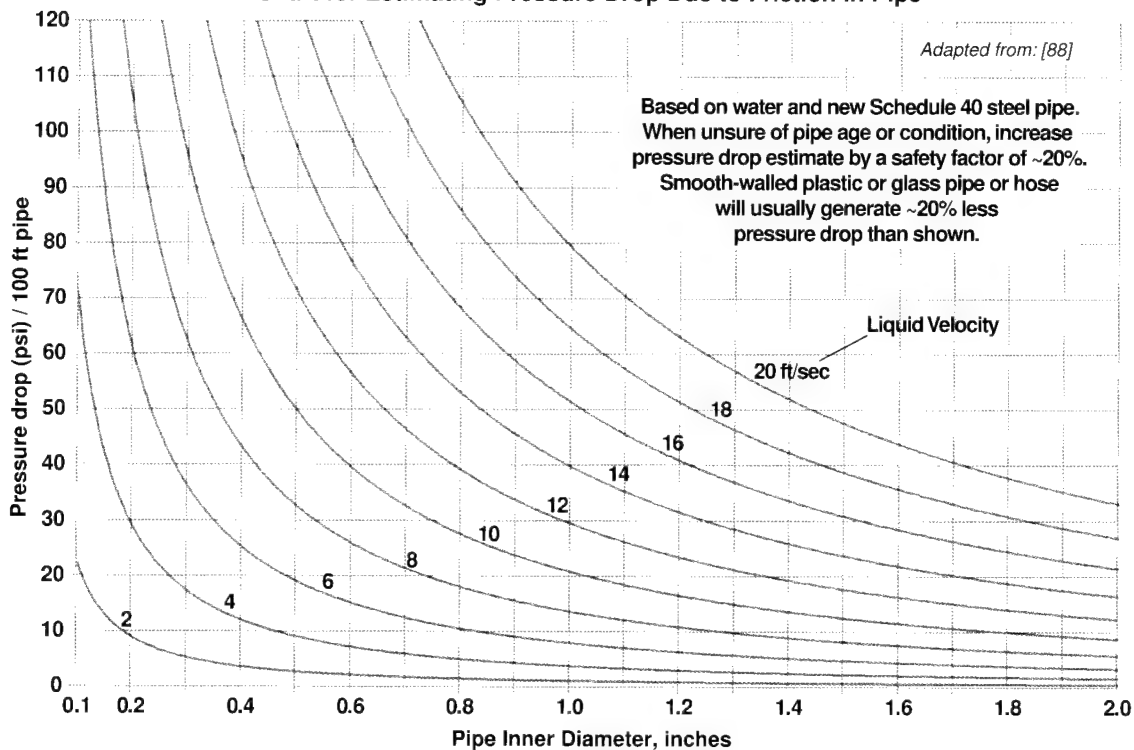






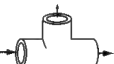


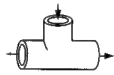




Chart for Estimating Pressure Drop Due to Friction in Pipe



Pressure Drop Through Pipe Fittings

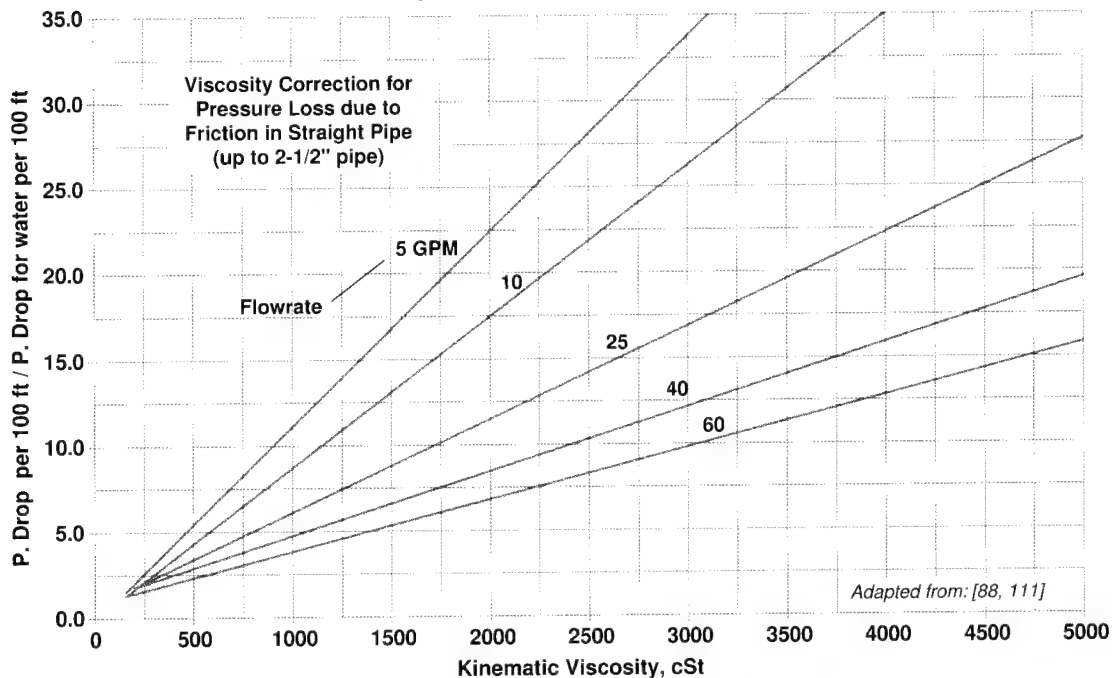
Fitting	Equivalent Length of Straight Pipe	Fitting	Equivalent Length of Straight Pipe	Fitting	Equivalent Length of Straight Pipe
elbow 	4 - 8 ft	coupling/union 	1 - 3 ft	90° angle valve 	15 - 20 ft
45° bend 	1 - 3 ft	180° bend 	1 - 3 ft	one-size reducer 	4 - 8 ft
tee-line flow 	2 - 8 ft	globe valve 	20 - 60 ft	flap check-valve 	8 - 12 ft
tee-branch flow 	4 - 12 ft	gate valve 	1 - 3 ft	ball check-valve 	15 - 25 ft

Adapted from: [88]

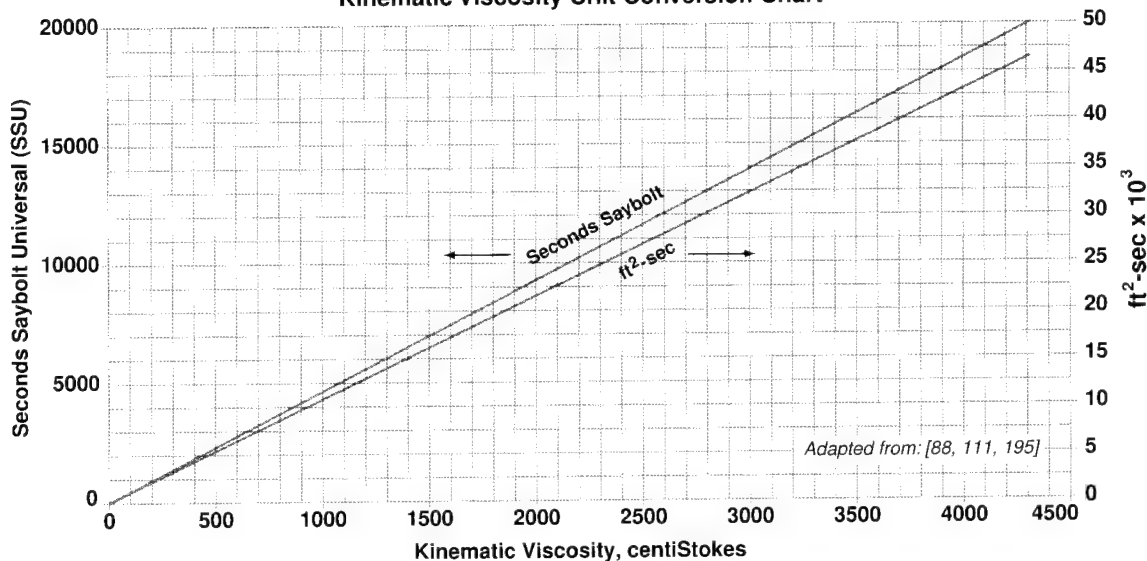
The table above lists the pressure drop experienced by water flowing through pipe fittings, expressed as equivalent feet of straight pipe of the same size. Use these numbers, together with the pressure loss chart at the top of the page, as a very rough guide when estimating total pressure drop in a piping system. A great deal of variation can be expected depending on the condition and material of the pipe and the flowrate. The range of numbers given above indicate the pressure drop that may be experienced with pipes ranging in size from 1/2" to 2". Pressure drop increases with larger pipe sizes. The table and the pressure-drop chart assume turbulent flow (Reynold's number > 2000). The pressure losses will be much lower at fluid velocities below about 1 ft/sec.

Viscosity Effects

Viscosity Correction for Pressure Loss in Pipe



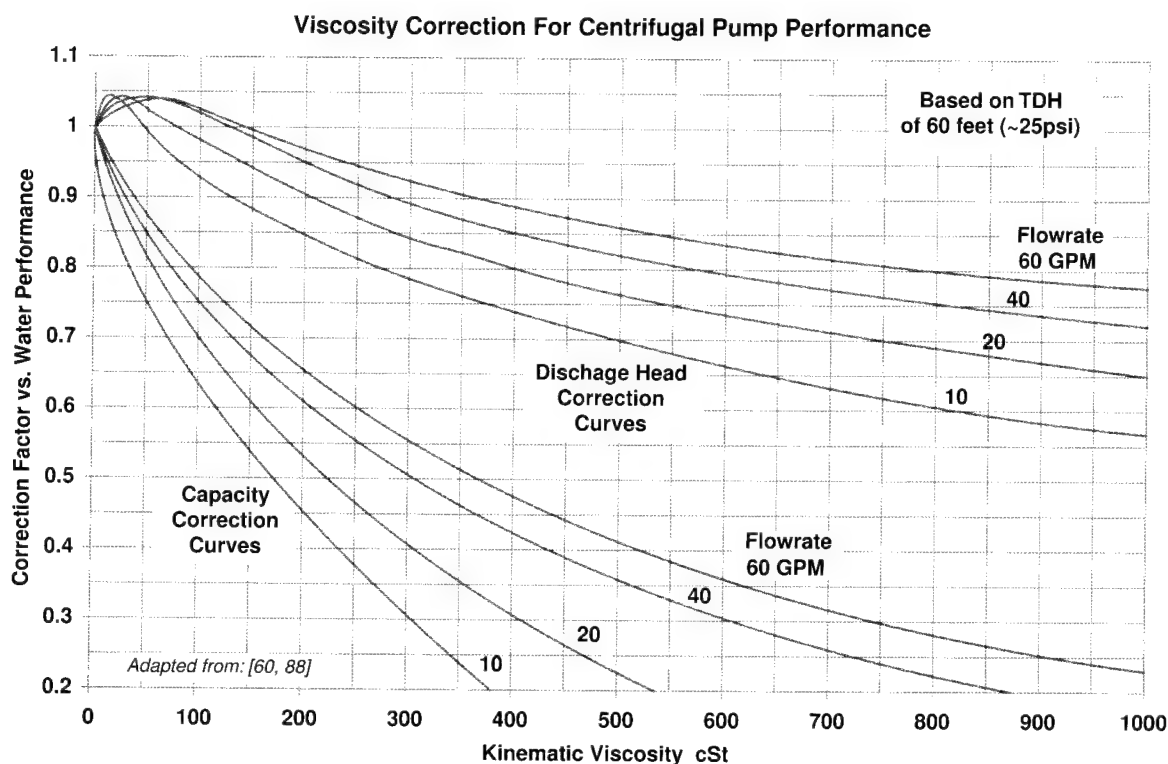
Kinematic Viscosity Unit Conversion Chart



Approximate Absolute and Kinematic Viscosity of Some Liquids

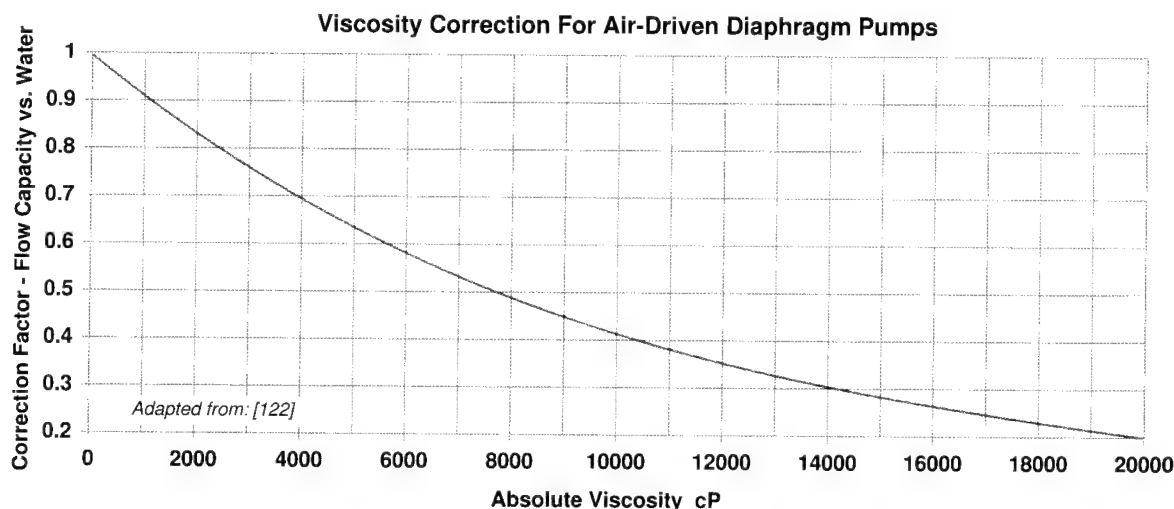
hexane	0.47 cP	0.7 cSt	butanediol	70 cP	69 cSt
acetone	0.6	0.8	50% NaOH	140	92
water	1	1	petroleum oil	300	350
milk	3	2.7	cake batter	2500	2300
toluene	5	6	liquid soap	3000	2500
blood plasma	20	16	honey	5500	4000
vegetable oil	30	35	molasses	7000	4500
ethylene glycol	60	54	peanut butter	30,000	~30,000

The viscosity of most liquids handled in the CPI will not exceed about 500 cP, except perhaps at very low temperatures. The table at the left is intended to provide a feel for the meaning of the viscosity values by comparing absolute and kinematic viscosities of some familiar liquids. Source [88].



The chart above shows the effect of fluid viscosity on small centrifugal pump performance, in terms of both capacity and discharge head. As viscosity increases, the percentage of the rated flow (based on water) that is actually realized decreases, as does the pump discharge pressure. Note that the effect of viscosity is greater at lower flowrates. The impact on discharge pressure is less severe, and in fact, discharge pressure first increases slightly before beginning to decrease. The correction factors are also a function of total dynamic head, and again, at lower TDH, the viscosity effect is greater. As a very rough guide, assume that doubling the TDH decreases the correction factor by about 10%, halving it increases the correction factor by about 10% (i.e., multiple factor from chart by 1.10).

The chart below similarly shows the effect of absolute viscosity on flowrate for air-driven diaphragm pumps. Since diaphragm pumps are positive displacement, the effect of TDH and flowrate on the correction factor are not significant. The effect on most other types of positive displacement pumps will be similar but less pronounced.



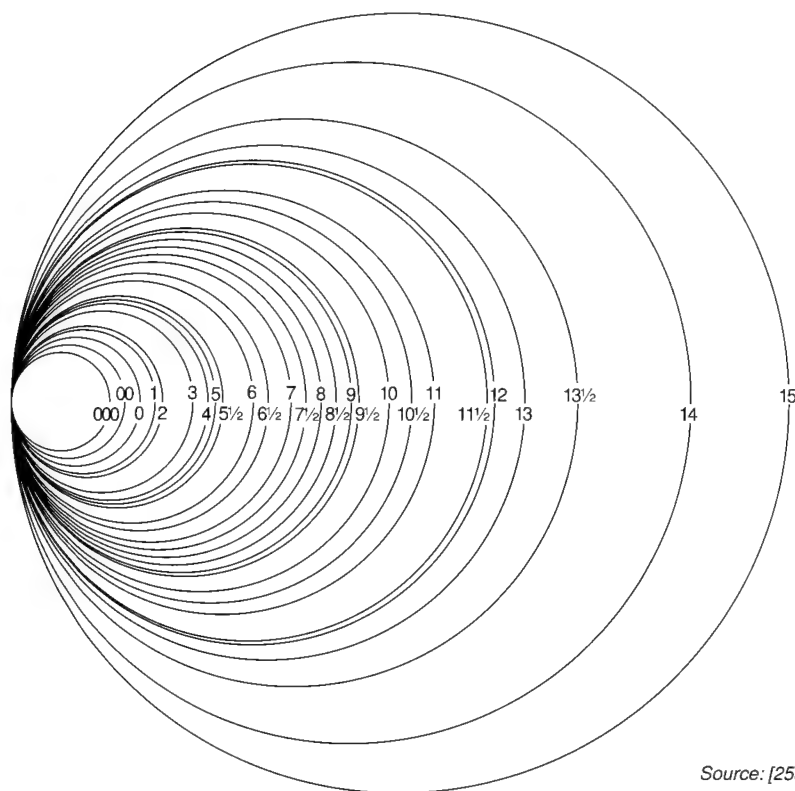
Standard O-Ring Size Chart

Dash No.	nom OD"	Dash No.	nom OD"	Dash No.	nom OD"	Dash No.	nom OD"	Dash No.	nom OD"
1/16" x-section		-109	1/2	-167	7-3/16	-248	5	-334	3
-003	3/16	-110	9/16	-168	7-7/16	-249	5-1/8	-335	3-1/8
-004	13/64	-111	5/8	-169	7-11/16	-250	5-1/4	-336	3-1/4
-005	7/32	-112	11/16	-170	7-15/16	-251	5-3/8	-337	3-3/8
-006	1/4	-113	3/4	-171	8-3/16	-252	5-1/2	-338	3-1/2
-007	9/32	-114	13/16	-172	8-7/16	-253	5-5/8	-339	3-5/8
-008	5/16	-115	7/8	-173	8-11/16	-254	5-3/4	-340	3-3/4
-009	11/32	-116	15/16	-174	8-15/16	-255	5-7/8	-341	3-7/8
-010	3/8	-117	1	-175	9-3/16	-256	6	-342	4
-011	7/16	-118	1-1/16	1/8" x-section		-257	6-1/8	-343	4-1/8
-012	1/2	-119	1-1/8	-201	7/16	-258	6-1/4	-344	4-1/4
-013	9/16	-120	1-3/16	-202	1/2	-259	6-1/2	-345	4-3/8
-014	5/8	-121	1-1/4	-203	9/16	-260	6-3/4	-346	4-1/2
-015	11/16	-122	1-5/16	-204	5/8	-261	7	-347	4-5/8
-016	3/4	-123	1-3/8	-205	11/16	-262	7-1/4	-348	4-3/4
-017	13/16	-124	1-7/16	-206	3/4	-263	7-1/2	-349	4-7/8
-018	7/8	-125	1-1/2	-207	13/16	-264	7-3/4	-350	5
-019	15/16	-126	1-9/16	-208	7/8	-265	8	-351	5-1/8
-020	1	-127	1-5/8	-209	15/16	-266	8-1/4	-352	5-1/4
-021	1-1/16	-128	1-11/16	-210	1	-267	8-1/2	-353	5-3/8
-022	1-1/8	-129	1-3/4	-211	1-1/16	-268	8-3/4	-354	5-1/2
-023	1-3/16	-130	1-13/16	-212	1-1/8	-269	9	-355	5-5/8
-024	1-1/4	-131	1-7/8	-213	1-3/16	-270	9-1/4	-356	5-3/4
-025	1-5/16	-132	1-15/16	-214	1-1/4	-271	9-1/2	-357	5-7/8
-026	1-3/8	-133	2	-215	1-5/16	-272	9-3/4	-358	6
-027	1-7/16	-134	2-1/16	-216	1-3/8	-273	10	-359	6-1/8
-028	1-1/2	-135	2-1/8	-217	1-7/16	-274	10-1/4	-360	6-1/4
-029	1-5/8	-136	2-3/16	-218	1-1/2	-275	10-3/4	-361	6-3/8
-030	1-3/4	-137	2-1/4	-219	1-9/16	-276	11-1/4	-362	6-5/8
-031	1-7/8	-138	2-5/16	-220	1-5/8	-277	11-3/4	-363	6-7/8
-032	2	-139	2-3/8	-221	1-11/16	-278	12-1/4	-364	7-1/8
-033	2-1/8	-140	2-7/16	-222	1-3/4	3/16" x-section		-365	7-3/8
-034	2-1/4	-141	2-1/2	-223	1-7/8	-309	13/16	-366	7-5/8
-035	2-3/8	-142	2-9/16	-224	2	-310	7/8	-367	7-7/8
-036	2-1/2	-143	2-5/8	-225	2-1/8	-311	15/16	-368	8-1/8
-037	2-5/8	-144	2-11/16	-226	2-1/4	-312	1	-369	8-3/8
-038	2-3/4	-145	2-3/4	-227	2-3/8	-313	1-1/16	-370	8-5/8
-039	2-7/8	-146	2-13/16	-228	2-1/2	-314	1-1/8	-371	8-7/8
-040	3	-147	2-7/8	-229	2-5/8	-315	1-3/6	-372	9-1/8
-041	3-1/8	-148	2-15/16	-230	2-3/4	-316	1-1/4	-373	9-3/8
-042	3-3/8	-149	3	-231	2-7/8	-317	1-5/16	-374	9-5/8
-043	3-5/8	-150	3-1/16	-232	3	-318	1-3/8	-375	9-7/8
-044	3-7/8	-151	3-3/16	-233	3-1/8	-319	1-7/16	-376	10-1/8
-045	4-1/8	-152	3-7/16	-234	3-1/4	-320	1-1/2	-377	10-3/8
-046	4-3/8	-153	3-11/16	-235	3-3/8	-321	1-9/16	-378	10-7/8
-047	4-5/8	-154	3-15/16	-236	3-1/2	-322	1-5/8	-379	11-3/8
-048	4-7/8	-155	4-3/16	-237	3-5/8	-323	1-11/16	-380	11-7/8
-049	5-1/8	-156	4-7/16	-238	3-3/4	-324	1-3/4	-381	12-3/8
-050	5-3/8	-157	4-11/16	-239	3-7/8	-325	1-7/8	1/4" x-section	
3/32" x-section		-158	4-15/16	-240	4	-326	2	-425	5
-102	1/4	-159	5-3/16	-241	4-1/8	-327	2-1/8	-426	5-1/8
-103	9/32	-160	5-7/16	-242	4-1/4	-328	2-1/4	-427	5-1/4
-104	5/16	-161	5-11/16	-243	4-3/8	-329	2-3/8	-428	5-3/8
-105	11/32	-162	5-15/16	-244	4-1/2	-330	2-1/2	-429	5-1/2
-106	3/8	-163	6-3/16	-245	4-5/8	-331	2-5/8	-430	5-5/8
-107	13/32	-164	6-7/16	-246	4-3/4	-332	2-3/4	-431	5-3/4
-108	7/16	-165	6-11/16	-247	4-7/8	-333	2-7/8	-432	5-7/8
		-166	6-15/16						

Adapted from Parker-Hannifin [193]

Rubber Stopper Sizes

Rubber Stopper Sizing Diagram (Top Diameter)



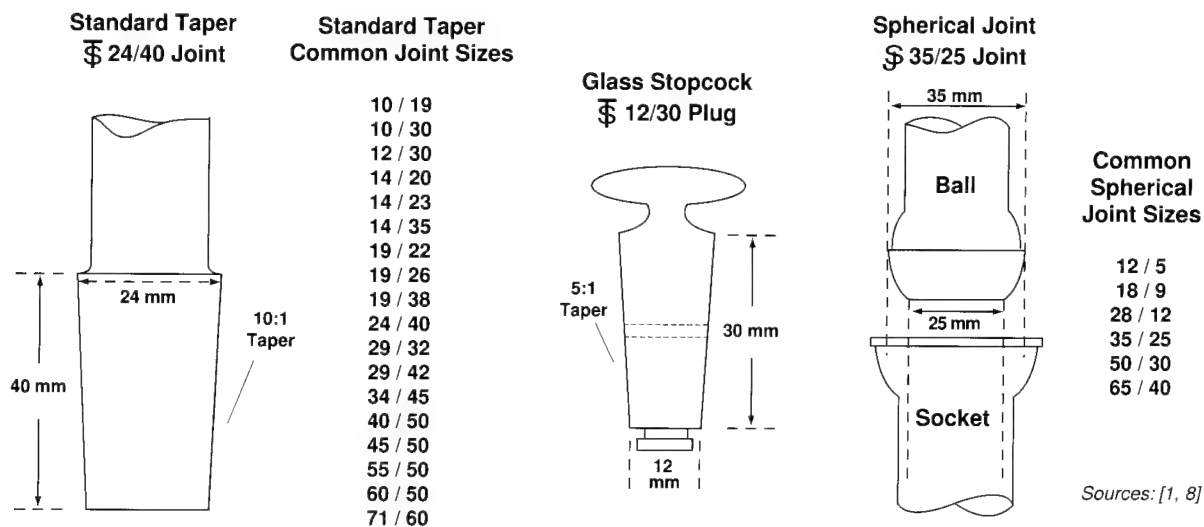
Source: [253]

Rubber Stopper Dimensions

Size	Top Diam. mm	Bot. Diam. mm	Length mm
000	13	8	21
00	15	10	25
0	17	13	25
1	19	14	25
2	20	16	25
3	24	18	25
4	26	20	25
5	27	23	25
5½	28	24	25
6	32	26	25
6½	34	27	25
7	37	30	25
7½	39	31	25
8	41	33	25
8½	43	36	25
9	45	37	25
9½	46	38	25
10	50	42	25
10½	53	45	25
11	56	48	25
11½	63	50	25
12	64	54	25
13	68	58	25
13½	75	62	35
14	90	75	39
15	103	83	39

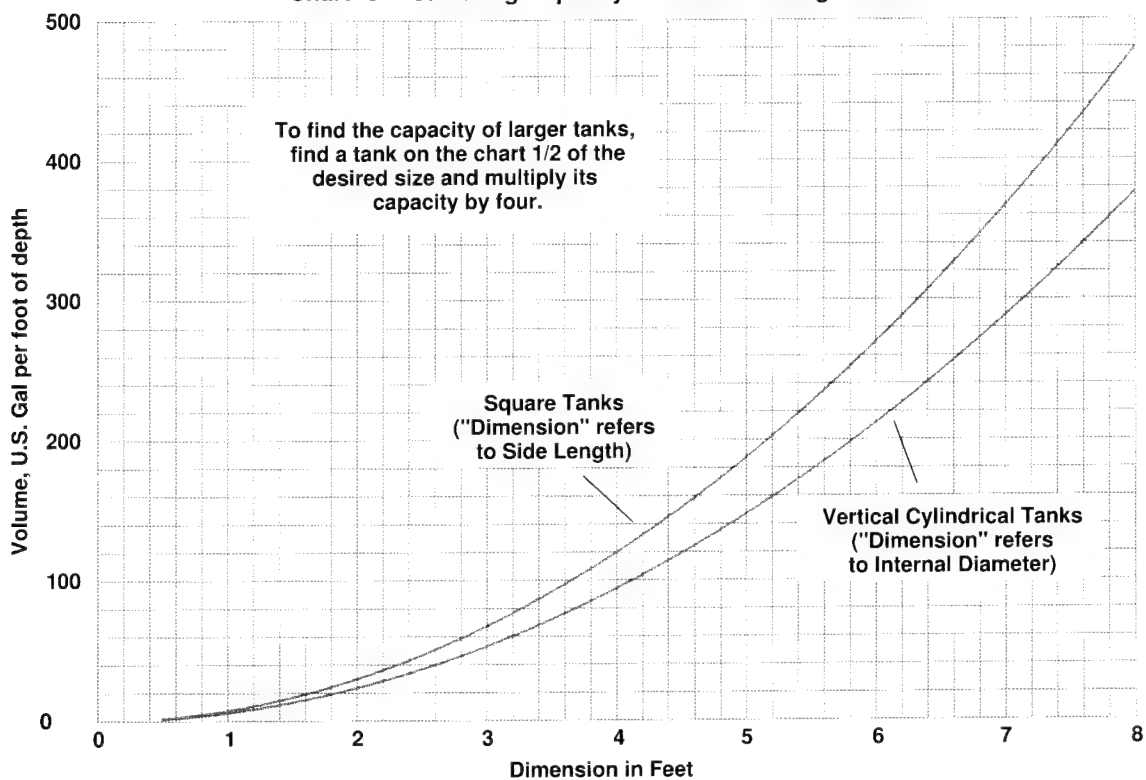
Ground Glass Joint Sizes

The figure below shows the method for determining ground glass joint dimensions and lists the most commonly used sizes for both standard tapered joints, stopcocks and spherical joints (usually used for laboratory high vacuum service). A 10:1 taper angle is standard for ground-glass joints. Tapered stopcocks use a 5:1 taper. Ground glass fittings should be sealed and lubricated with silicone or other high-temperature/high vacuum grease, especially for vacuum applications.

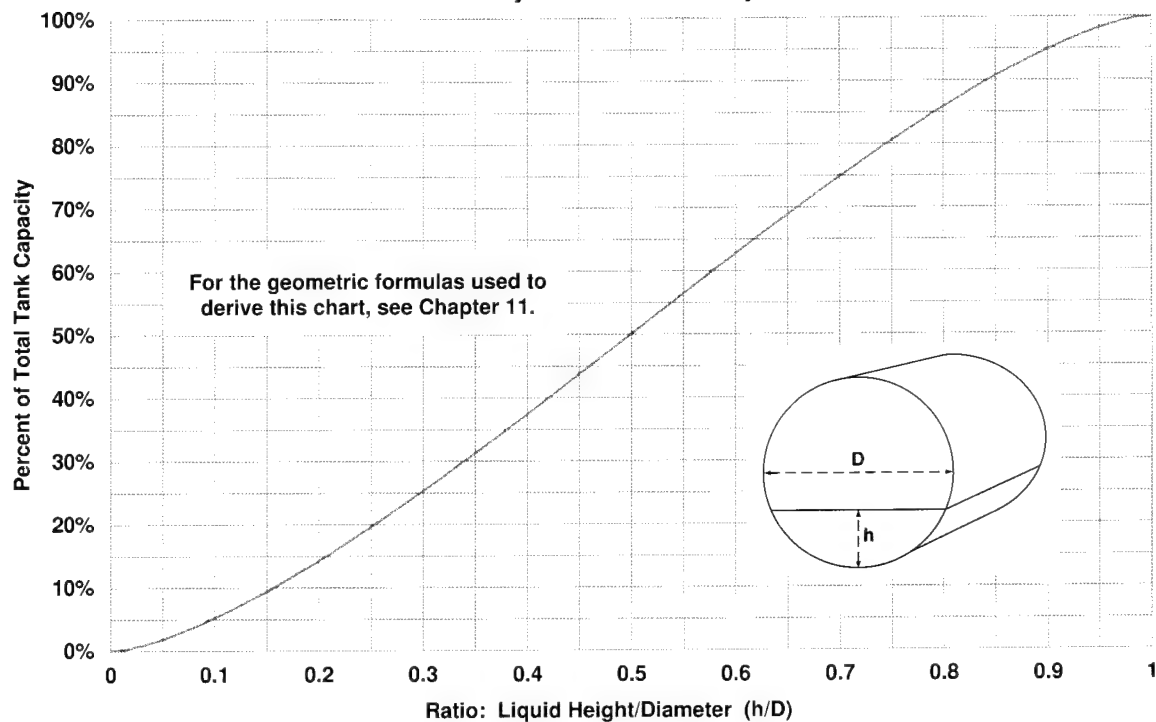


Capacity of Liquid Storage Tanks

Chart for Estimating Capacity of Vertical Storage Tanks

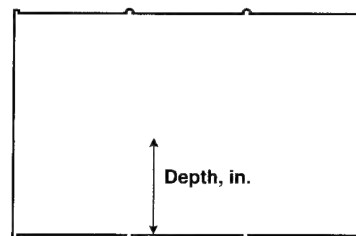
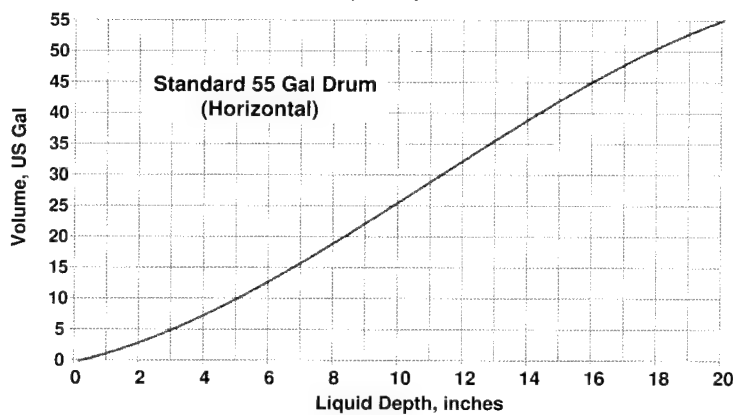
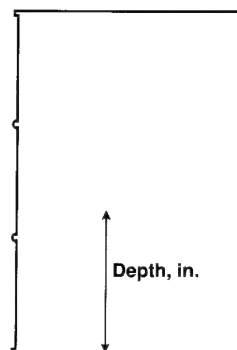
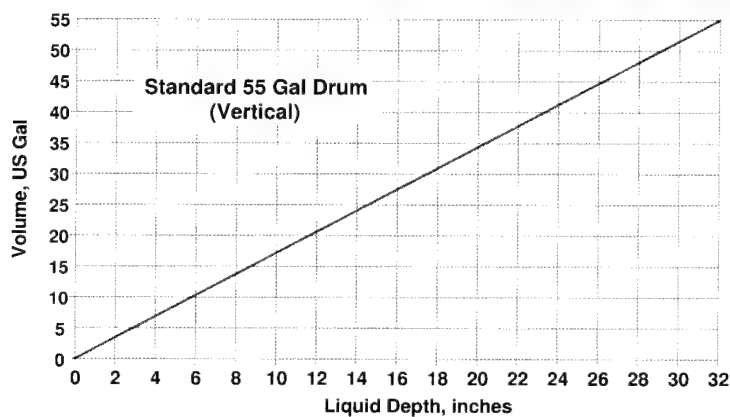


Volume of Partially Filled Horizontal Cylindrical Tanks

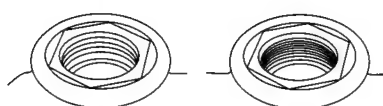


Storage Drum and Shuttle Data

Charts for Estimating Drum Contents

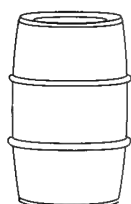


Common Closure Styles on Various Drums, Totes and Liquid Shuttles

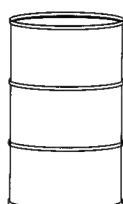


2" Buttress
Tank Opening

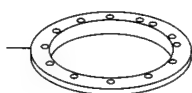
2" NPT Female



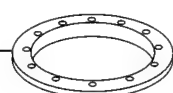
55-Gal
Poly Drum



55-Gal
Metal Drum



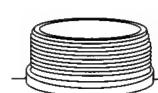
6" (15cm)
DD6 Opening
(6- or 13-bolt)



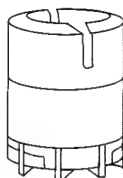
6" (15cm)
12-bolt
Opening



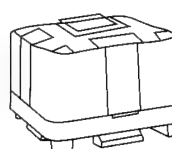
4" (10cm)
Threaded
GPI Opening



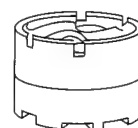
Snyder
Gem Cap
Thread



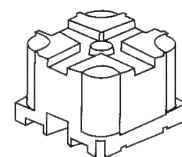
Dow, Snyder
New Gen. Totes



Cyanamid
System 110



Monsanto
Shuttle



Square
Stackable

Others include Novartis Field-Pak and Farm-Pak, and Zececa E-Z Handler. Totes such as these range in size from 200-800 gal and are moved by forklift.

4 Heat Transfer

Contents

REACTORS

Heat Transfer in Stirred Tank Reactors	4-2
Typical Batch Cooling Profiles	4-4
Reactor Heat Transfer Coefficients	4-5
Experimental Determination of Heat Transfer Coefficient	4-7

HEAT EXCHANGERS

Heat Exchanger Fundamentals	4-8
Example Heat Exchanger Problem	4-9
Chart for Estimating Log Mean Temperature Difference	4-10

TEMPERATURE CONTROL UNITS

Temperature Control Units	4-11
System Start-up and Operation	4-12
Tips on Using Heat Transfer Fluids in TCUs	4-13

HEAT TRANSFER FLUIDS

Heat Transfer Fluid Selection	4-14
Specific Heat of Various Heat Transfer Fluids vs. Temperature	4-15
Useful Temperature Ranges and Properties of Commercial HTFs	4-16
Notes on the Use of Glycol HTFs	4-18
Specific Gravity of Aqueous Glycol Solutions	4-19
Freezing Points of Aqueous Glycol Solutions	4-20
Boiling Points of Aqueous Glycol Solutions	4-21
Specific Heats of Aqueous Glycol Solutions	4-22
Viscosity of Aqueous Ethylene Glycol Solutions	4-23
Freezing Points of Brines	4-24
Steam Pressure vs. Temperature	4-25

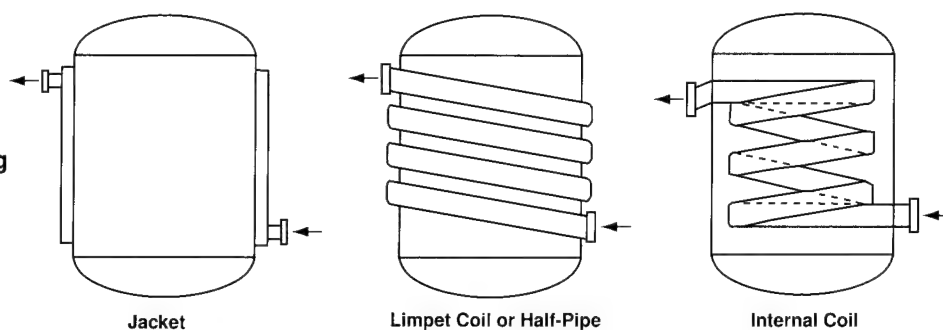
PROCESS CHILLERS

Notes on Process Chillers	4-26
Approximate Chiller Capacity	4-28
Effect of Condenser Cooling Water Temperature	4-28

Heat Transfer in Stirred Tank Reactors

Heating and cooling agitated vessels is usually accomplished by injecting steam or circulating a heat transfer fluid through the jacket or heating coils. Simple **external jackets** are the most common for multipurpose reactor vessels in the fine chemical industry. Often the jackets are dimpled or baffled to improve their relatively poor heat transfer performance. **Internal coils** can be more economical to install and offer improved heat transfer characteristics and higher operating pressures than jackets, but may not be useful for certain applications such as mixing viscous liquids and crystal slurries. **External coils** (called limpet coils or half-pipe jackets) allow for better distribution of heat than simple jackets, but can be difficult to construct and often leak. The figures below illustrate these three major configurations. Split jackets consist of separate upper and lower sections to enable heating small volumes without baking material to the inside upper surfaces.

Common Vessel Heating and Cooling Arrangements



Vessel Heat Transfer Calculations – Calculating rates of heating and cooling is fairly straightforward as long as the necessary information about the batch and the vessel is available. In the case of small simple jacketed reactors, the flowrate of heat transfer fluid through the jacket is usually high enough that there is not a significant difference between the inlet and outlet temperatures, i.e., jacket temperature can be considered constant. This allows us to use the equation for heating or cooling with an isothermal heat transfer medium, which is somewhat simpler to use than the equations for the non-isothermal case.

The simple example below serves to illustrate the use of this equation. Further information on this and other relationships describing the behavior of various reactor configurations, including non-isothermal heating and cooling, is available in a number of excellent texts, including [60, 120, 266].

Vessel Cooling Example

How long will it take to cool a 50 gal jacketed vessel of water ($M = 410 \text{ lbm}$, $C_p = 1.0 \text{ Btu/lbm}^\circ\text{F}$) from an initial temperature, T_0 of 100°C (212°F) to a final temperature, T of 50°C (122°F), assuming an isothermal jacket temperature (T_J) of 20°C (68°F). The heat transfer surface area, A , is 8.1 ft^2 and the overall heat transfer coefficient, U , is $59.0 \text{ Btu/hr ft}^2 \text{ }^\circ\text{F}$. For isothermal cooling conditions, the following relationship can be used [60]:

$$\ln \frac{T_J - T_0}{T_J - T} = \left(\frac{UA}{MC_p} \right) t$$

Thus,

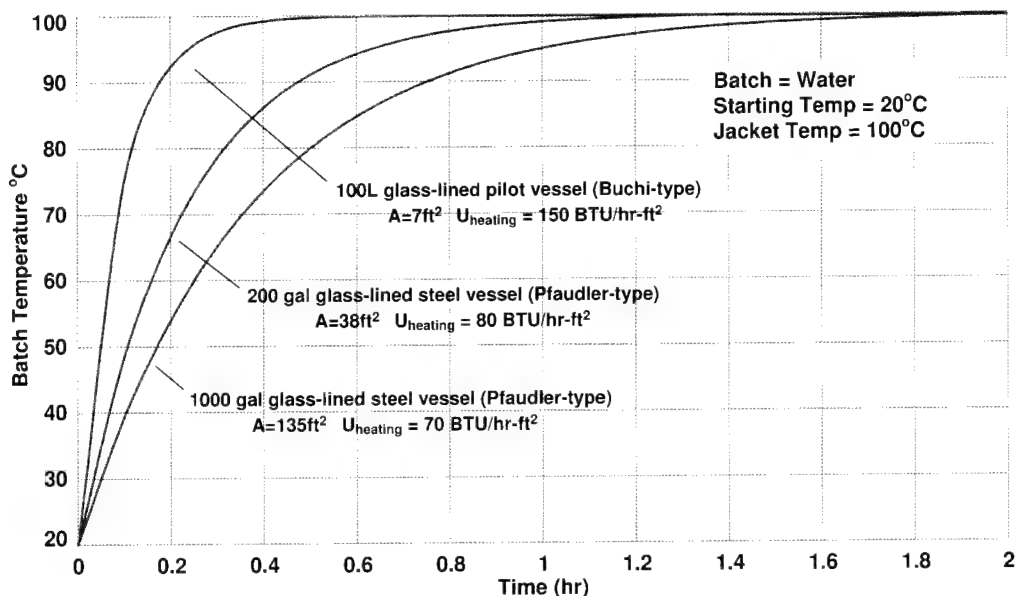
$$t = \ln \frac{T_J - T_0}{T_J - T} \times \frac{MC_p}{UA}$$

$$t = \ln \frac{68 - 212}{68 - 122} \times \frac{410 \text{ lbm} \times 1.0 \text{ Btu/lb}^\circ\text{F}}{59.0 \text{ Btu/hr ft}^2 \text{ }^\circ\text{F} \times 8.1 \text{ ft}^2}$$

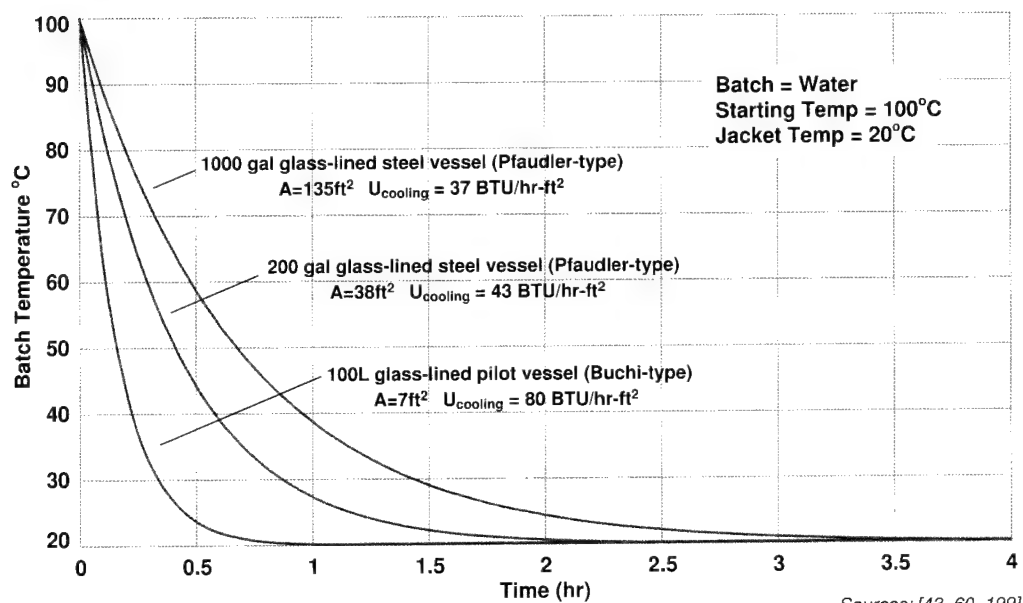
$$t = 0.98 \times 0.86 \text{ hr} = 0.84 \text{ hr}$$

Metric units may also be used, as long as they are used consistently. Mass must be in kg, C_p in $\text{kJ/kg}\cdot\text{K}$, T in $^\circ\text{C}$ or K , A in m^2 , U in $\text{W/m}^2\cdot\text{K}$. Use the conversion factor $1 \text{ W} = 1 \text{ J/sec}$. The isothermal jacket assumption is a simplification which ignores startup effects, but allows a reasonable first estimate.

Typical Theoretical Batch Heating Profiles



Typical Theoretical Batch Cooling Profiles

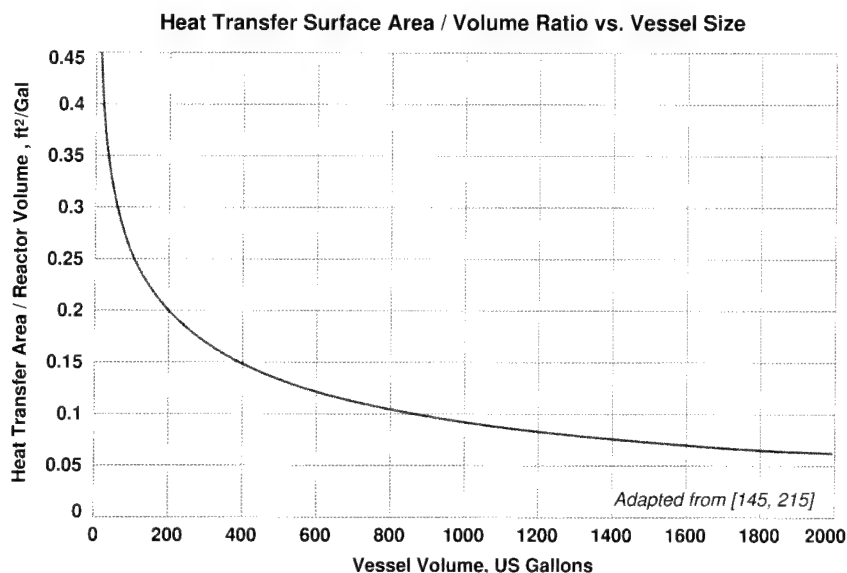


Sources: [43, 60, 199]

The graphs above show the theoretical heating and cooling rates of some typical reactors given the starting batch and jacket temperatures shown. The curves were calculated using the isothermal heat transfer equation applied in the example on the previous page, and thus represent the maximum possible rates of heating and cooling. Certain important factors are ignored, such as the time required to bring the jacket up to temperature. Also, in practice, the temperature is often ramped by a programmable TCU to maintain a certain profile during heating or cooling, and thus longer times may be required. Other limitations might include the maximum temperature differential between batch and jacket to which the reactor can be exposed, especially for glass-lined reactors. Nonetheless, the graphs illustrate the basic point that larger reactors take longer to heat or cool than smaller ones, even if the jacket temperature is the same.

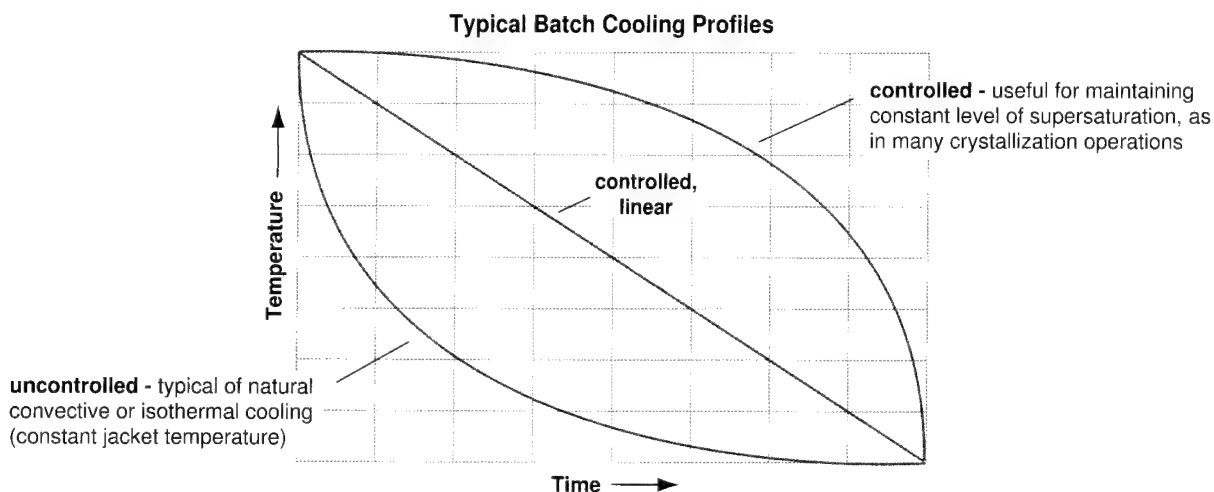
Two factors account for these differences in heating and cooling rates. One is the fact that larger reactors have less

available heat transfer surface area per unit volume than smaller reactors. This is because reactor volume increases as the cube of vessel size, whereas surface area increase only as the square of vessel size. This is illustrated in the graph below. Large industrial reactors often have less than 1/10 the available area per unit volume as bench reactors.



The second major reason for heating and cooling rate differences is the value of the heat transfer coefficient, which can vary tremendously depending on the reactor material and the nature of the batch and heat transfer medium. This is discussed in more detail on the following page.

Typical Cooling Profiles – The graph below depicts three typical types of batch cooling profiles encountered in chemical processing. The most common is the uncontrolled, or natural, cooling profile, wherein the vessel and its contents are either allowed to cool to ambient temperature naturally without forced convection, or are cooled by circulating a cold fluid of relatively fixed temperature through vessel jacket or coil. This is another example of isothermal cooling conditions as described above. The highest rate of heat removal takes place during the earliest part of the cycle. The other types of profiles shown are controlled, that is, the coolant temperature and therefore the rate of heat removal is ramped in a controlled manner to accomplish a specific processing objective such as maintaining constant supersaturation during a crystallization or similar operation. Generally speaking, controlled cooling profiles can be scaled up much more successfully than uncontrolled.



Reactor Heat Transfer Coefficients

The reactor overall heat transfer coefficient (OHTC), or “U”, can be the most difficult element of the heat transfer equation to quantify. Values for agitated vessels typically range from about 100 to over 1500 W/m²-K (~20 to ~200 BTU/hr ft² °F) and are significantly affected by the heating medium characteristics, its flow pattern (jacket, coil, etc.) and flowrate, by the speed and type of agitation, by the condition of reactor surfaces and by the physical characteristics of the batch, such as viscosity, density, specific heat and thermal conductivity. Dependence on these factors means that, for typical reactors, OHTCs tend to be higher for heating than for cooling. There is also usually a significant lag time in batch heating or cooling, due to the time it takes to adjust the temperature of the thermal mass of the reactor vessel, the piping system, and the heat transfer fluid itself. This means that the value of U will likely change throughout the heating or cooling operation. The two tables below show typical ranges of OHTC values for agitated vessels of various types and for glass-lined vessels under various heat transfer conditions. For the reasons listed above, values are approximate.

Typical Overall Heat Transfer Coefficients for Stirred Tank Reactors

Reactor Type	Heating		Cooling	
	W/m ² -K	Btu/hr ft ² °F	W/m ² -K	Btu/hr ft ² °F
Simple Jacket (Mild Steel Vessel)	400-900	70-160	150-600	25-105
Simple Jacket (Glass-lined Vessel)	200-700	35-125	100-350	20-60
Dimpled Jacket	500-1000	90-175	300-550	50-100
Limpet Coil	600-1100	105-190	200-700	35-125
Internal Coil	600-1500	105-260	250-800	45-140

1 Btu/hr ft²°F = 5.678 W/m²-K

Sources: [60, 97, 120, 125, 215, 222]

Typical Overall Heat Transfer Coefficients for Glass-Lined Reactors

Heat Transfer Conditions	W/m ² -K	Btu/hr ft ² °F
Water, heated with steam	440	77
Water, heated with synthetic HTF	320	56
Organic solvent cooled with water	190	34
Viscous organic cooled with water	100	18

Sources: [97, 138, 199]

Estimating Heat Transfer Coefficient – If the key batch properties are known and enough information about the vessel geometry is available, a reasonable estimate of overall U can be made using empirical relationships, such as the one first described by Chilton [58]. A simplification of this equation (assuming all fluid properties and jacket temperature remain constant) is used in the example below. The graphical representation of the equation on the following page is also useful. The value “a” in the equation is an empirical mixing factor that accounts for differences in agitator, jacket and coil design. Typical values of “a” are also shown on the following page.

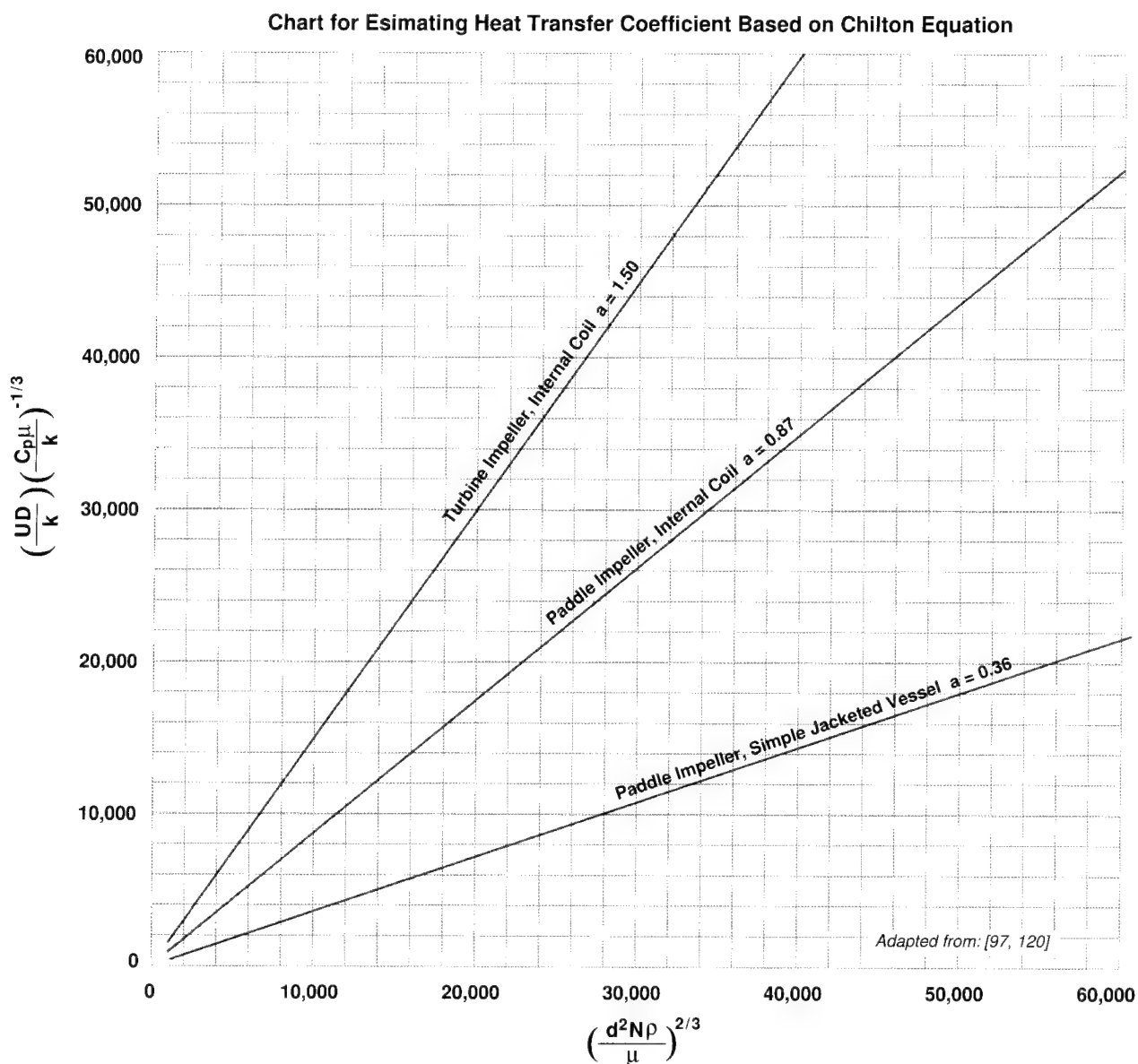
Heat Transfer Coefficient Estimation Example

A vessel with an internal coil has a diameter D=8 ft, a paddle-style agitator (a=0.87) with diameter d=4 ft, turning at N=12000 rev/hr. The batch fluid has a density of ρ=40 lb/ft³, viscosity of μ=4.0 lb/ft-hr, thermal conductivity of k=0.10 Btu/hr-ft-°F, and specific heat Cp=0.80 Btu/lb-°F. Estimate the overall heat transfer coefficient. The following equation may be applied [120]:

$$\left(\frac{UD}{k}\right) = a \left(\frac{d^2 N \rho}{\mu}\right)^{2/3} \left(\frac{C_p \mu}{k}\right)^{1/3}$$

The three terms in this equation represent the following dimensionless groups: (UD/k) = the Nusselt number [N_{Nu}], ($d^2 N \rho / \mu$) = the mixing Reynold's number [N_{Re}] and ($C_p \mu / k$) = the Prandtl number [N_{Pr}]. Substituting the values given, ($d^2 N \rho / \mu$)^{2/3} = 15,448 and ($C_p \mu / k$)^{1/3} = 3.175. Thus U=533 Btu/hr-ft²-°F (3026 W/m²-°K).

Alternatively, after calculating the value of ($d^2 N \rho / \mu$)^{2/3} the chart on page 4-6 could have been used to estimate the value of (UD/k)($C_p \mu / k$)^{-1/3} by reading off the line for a = 0.87, and then solving for U.



The chart above is a graphical representation of the Chilton equation used in the example on the previous page. It can be a useful way of estimating U if the specifics of the reactor and the physical characteristics of the batch are known, and can simplify the somewhat rigorous calculations required when solving the equation. When performing any such engineering calculations, it should go without saying that consistent use and proper cancellation of units are critical to obtaining a correct result.

Typical "a" (Mixing Factor) Values

Agitator Type	Jacketed	Internal Coil
Turbine	0.62	1.50
Paddle	0.36	0.87
Propeller	0.54	0.83
Anchor	0.46	-

Sources: [97, 120]

The Chilton equation is also useful in estimating the effect of a change in batch characteristics on the overall heat transfer coefficient. By way of example, the equation indicates that U is expected to vary as specific heat to the $1/3$ power. Thus, doubling the specific heat of the batch, keeping all other things equal, should result in an approximately 26% increase in the value of U , since $2^{1/3} = 1.26$. This is only a theoretical result, since, in fact, many batch parameters work synergistically in ways not well described by the equation.

Experimental Determination of Heat Transfer Coefficient

Theoretical estimates and literature values of the heat transfer coefficient are of limited utility. As described above, so many variables can impact heat transfer rates that values of U for any given reactor are best determined experimentally under controlled conditions. The measurement is fairly straightforward, as long as the instrumentation is in place to collect the necessary data, specifically jacket inlet, jacket outlet and batch temperature vs. time. These data, along with the physical properties of the batch, are used to derive the heat transfer coefficient using the isothermal heat transfer equation described earlier.

In order to arrive at U , an accurate value of the heat transfer area is necessary. However, determining the exact heat transfer area at partial volumes is not trivial. Nor is it strictly necessary. It is usually sufficient for most heat transfer calculations in batch reactors to know the value of the product UA . Using the same time/temperature data described above, the value for UA is easily obtained, as demonstrated in the example at the bottom of the page. In the example, temperature data are collected over time during a batch heating operation. The jacket temperature used is the average of the inlet and outlet temperatures. If isothermal conditions are assumed, then there should be no significant difference between inlet and outlet temperatures, but using the average is reasonable even if there is a marked difference. It will probably be necessary to ignore the first one or two data points, which represent the jacket warm-up period.

Since the value of UA will be different at different reactor volumes, it is important that the measurement be made at the expected batch operating volume, or better yet, at 3 to 5 different volumes, so that a chart of UA vs. reactor volume may be prepared. Ideally, this should be carried out for each vessel in the plant at the time of IQ/OQ (see page 2-7), but it can obviously be carried out at any time.

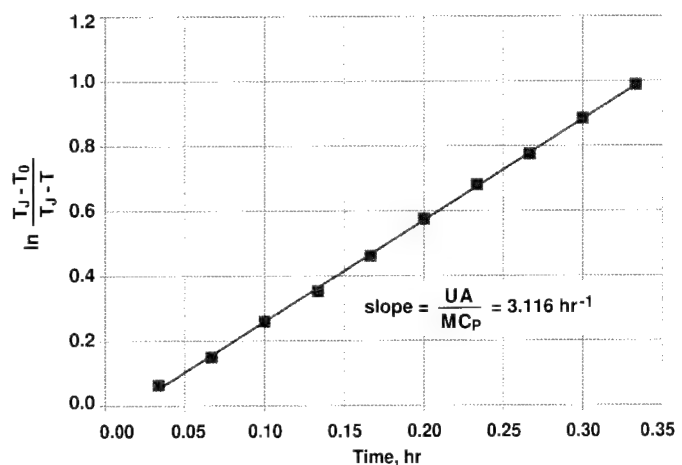
It is also important to remember that heat transfer coefficient, and the value UA , are strongly dependent on the properties of the batch, the age and condition of the jacket, the heat transfer medium and the mixing rate, as discussed earlier in this section. For the results to be meaningful, the measurement should be made as closely as possible to actual expected operating conditions.

Measurement of UA Example

During an operational test of a 30-gal Hastelloy reactor, the following time/temperature data were collected. The reactor was filled with water ($M=209$ lb, $C_p = 1.0$ Btu/lb-°F). Determine the value of UA at these conditions.

Time (min)	Time (hr)	Temp Batch °C	Avg. Temp Jacket °C	$\ln \frac{T_J - T_0}{T_J - T}$
0	0.00	20.0	20.0	-
2	0.03	22.5	60.6	0.064
4	0.07	25.6	60.2	0.150
6	0.10	29.2	60.2	0.260
8	0.13	31.9	60.0	0.353
10	0.17	34.8	60.0	0.462
12	0.20	37.4	59.8	0.575
14	0.23	39.9	60.3	0.681
16	0.27	41.8	60.4	0.776
18	0.30	43.6	60.2	0.884
20	0.33	45.3	60.3	0.988

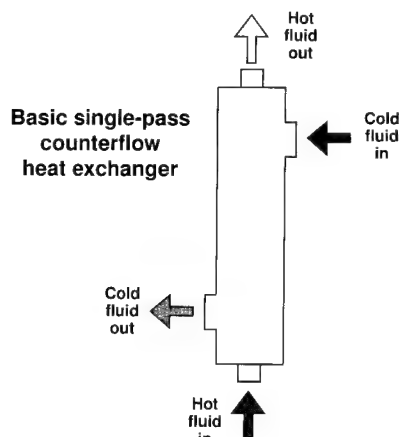
$$\ln \frac{T_J - T_0}{T_J - T} = \left(\frac{UA}{MC_p} \right) t$$



When the data are plotted as shown using the above isothermal heat transfer equation, linear regression gives a slope $UA/MC_p = 3.116$ hr⁻¹. Substituting the values for M and C_p , the value of UA is calculated as 651 Btu/hr °F. In this case the value of A was known to be 9.4 ft², and thus $U = 69.3$ Btu/hr ft² °F. However, even if A were not known, the value of the product UA is sufficient for heat transfer calculations. It is strongly recommended that this measurement be carried out at several volumes, and a chart of UA vs. volume be prepared for each vessel. Sources [60, 138]. Data from [222].

Heat Exchanger Fundamentals

A heat exchanger is any device used to heat or cool a fluid by imparting heat to, or extracting heat from, a second fluid without allowing the two fluids to directly contact each other. The fluids may be liquids or gases. Examples include automobile radiators, wherein air is used to cool the circulating engine coolant, and distillation condensers, in which cold water is used to cool distillate vapors to a temperature below their boiling point.



Heat exchangers are widely used throughout the CPI. There are many types, including shell and tube, plate and frame, and carbon block exchangers, and these are available in many configurations and materials. Heat exchangers are usually operated in one of two primary modes, cocurrent flow or countercurrent flow. The diagram at the left shows the operating mode for a simple single-pass countercurrent heat exchanger.

Heat exchanger operation can be described by the following relationships:

$$Q = FUA\Delta TLM$$

$$Q = [MC_p(T_{in} - T_{out})] \text{ for hot liquids being cooled}$$

$$Q = [MC_p(T_{out} - T_{in})] \text{ for cold fluids being heated}$$

where Q is the overall heat transfer rate, U is the overall heat transfer coefficient and A is the heat exchange surface area, ΔTLM is the *log mean temperature difference*, F is a correction factor, M is the mass flow rate, C_p is the

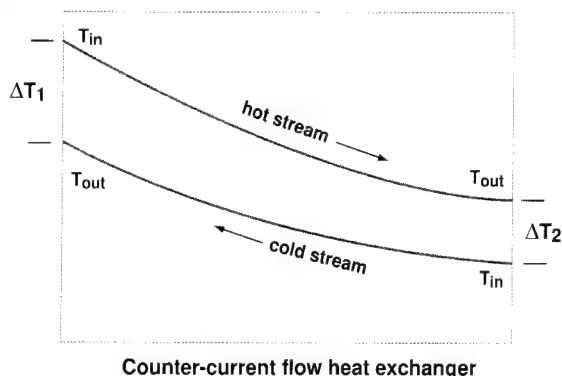
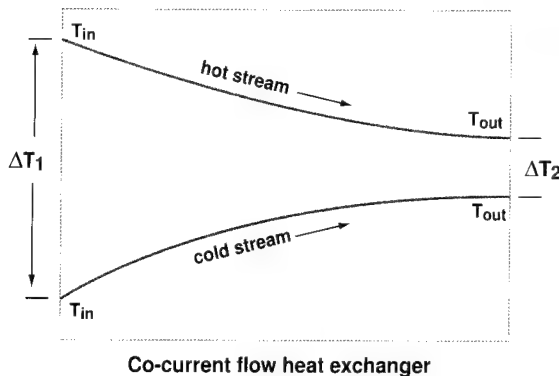
specific heat and $T_{in} - T_{out}$ or $T_{out} - T_{in}$ is the temperature difference accomplished by the heat exchanger for either of the phases. The factor F equals 1 for a simple countercurrent heat exchanger, but can vary from ~0.5 to ~1 for other configurations such as multipass units and heat exchange with phase change. Models for these other configurations are described in [120] and most good heat transfer texts.

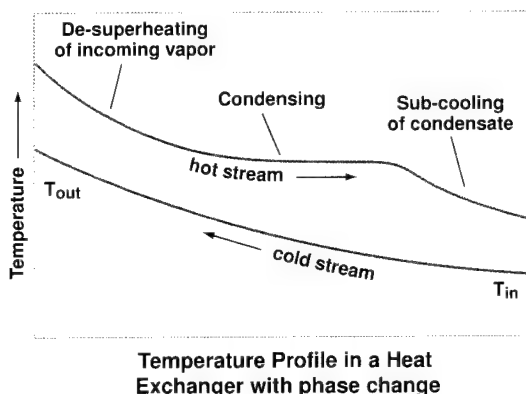
ΔTLM represents an "average" temperature difference throughout the heat exchanger based on the temperature differences of the entering and exiting streams. It is calculated according to the equation below but is defined somewhat differently for cocurrent and countercurrent flow heat exchangers (see the diagrams at the bottom of the page). The chart on page 4-10 can also be used to estimate ΔTLM .

$$\Delta TLM = \frac{\Delta T_1 - \Delta T_2}{\ln(\Delta T_1 / \Delta T_2)}$$

It should be kept in mind that these models assume that U and C_p remain constant and that the heat exchanger operates with 100% efficiency, whereas in reality, U may vary with location in the heat exchanger or with time

Typical Heat Exchanger Temperature Profiles





during the batch cycle, C_p is a function of temperature, and the condition and cleanliness of the exchanger surfaces can significantly affect the efficiency of the unit. The situation is even more complex in cases where a phase change of one or more of the streams takes place (see the figure at left). In this case, not only the temperature changes of all phases must be considered, but any enthalpies of vaporization, etc. that may be involved must be accounted for.

When estimating the required area for a heat exchanger, the heat duty Q and the ΔT values must be estimated based on expected process duty requirements, and any important fluid properties such as density and specific heat must be tabulated. This information can be provided to the heat exchanger manufacturer to

enable him to help you select the proper heat exchanger for your duty. Reasonably accurate values of U and necessary correction factors can be also obtained from any reputable manufacturer of heat exchangers.

The example below is provided to illustrate the application of the various heat exchanger equations mentioned here.

Example Heat Exchanger Problem

Oil at 320°F (160°C) enters a 237 ft² (22 m²) single pass counter-current heat exchanger and exits at 176°F (80°C). Cooling water enters at 68°F (20°C) at a flowrate of 23,810 lb/hr (3 kg/sec) and leaves at 158°F (70°C). Calculate the overall heat transfer coefficient, U . The specific heat of water at the mean temperature of 113°F (45°C) is 0.998 Btu/lbm°F (4180 J/kg°C).

First, based on the properties of the cold stream, solve for the heat transfer rate, Q :

$$Q = [MC_p(T_{out} - T_{in})]$$

$$23810 \frac{\text{lbm}}{\text{hr}} \times 0.998 \frac{\text{Btu}}{\text{lbm}^\circ\text{F}} \times (158 - 68) = 2,138,614 \frac{\text{Btu}}{\text{hr}}$$

$$\text{or} \quad 3 \frac{\text{kg}}{\text{sec}} \times 4180 \frac{\text{J}}{\text{kg}^\circ\text{C}} \times (70 - 20) = 627,000 \text{ W}$$

Then calculate the log mean temperature difference as follows (see the diagram at the bottom of page 4-8 for the definitions of ΔT_1 and ΔT_2 for co-current and counter-current heat exchangers:

$$\Delta T_{LM} = \frac{\Delta T_1 - \Delta T_2}{\ln(\Delta T_1 / \Delta T_2)} = \frac{162 - 108}{\ln(162 / 108)} = 133.2^\circ\text{F}$$

$$\text{or} \quad \Delta T_{LM} = \frac{90 - 60}{\ln(90 / 60)} = 74.0^\circ\text{C}$$

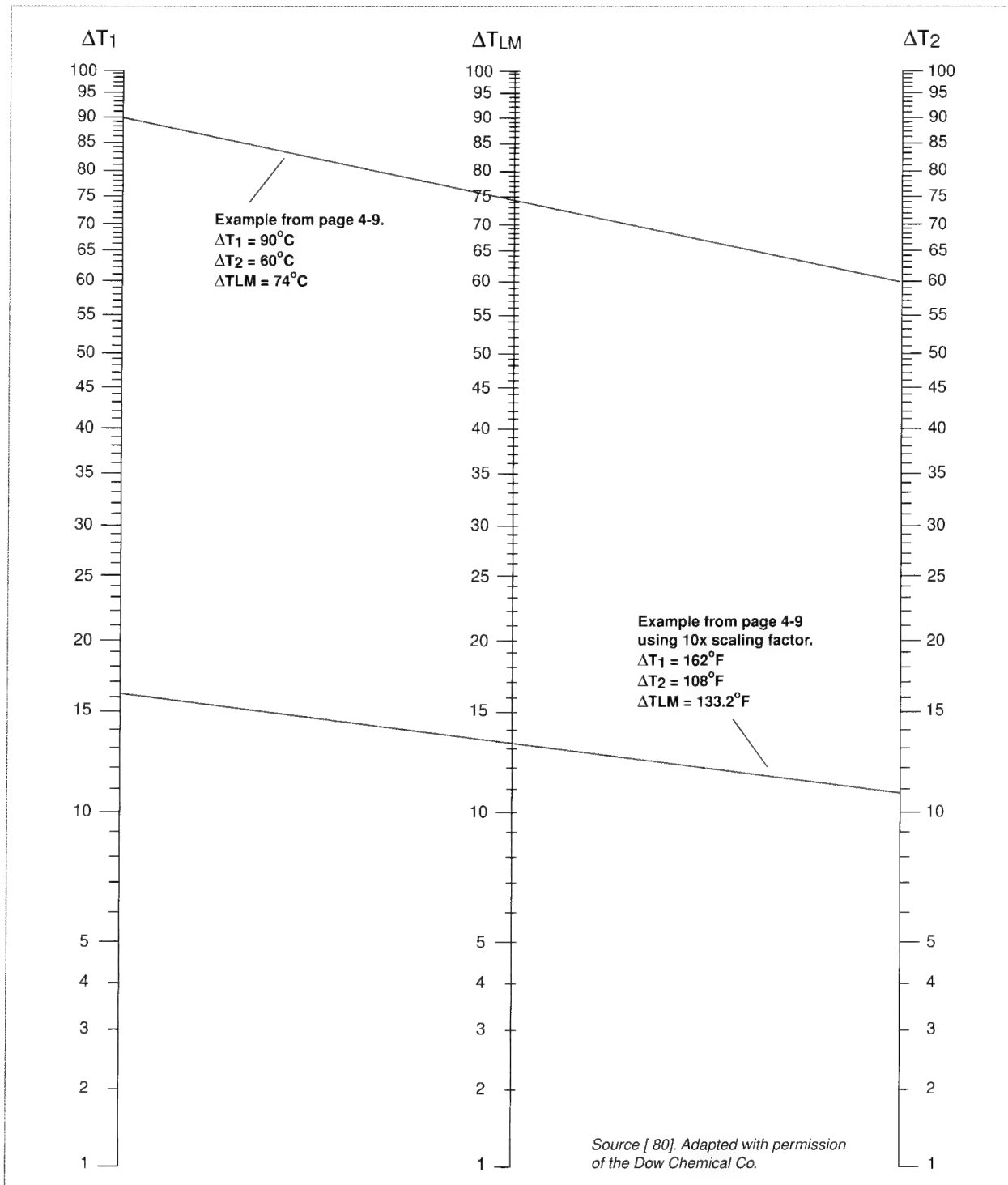
Finally, calculate the value of U (correction factor $F=1$ for single pass countercurrent flow):

$$Q = FUA\Delta T_{LM} \quad \text{or} \quad U = \frac{Q}{A\Delta T_{LM}}$$

$$U = \frac{2,138,614 \text{ BTU}}{\text{hr} \times 237 \text{ ft}^2 \times 133.2^\circ\text{F}} = 67.7 \text{ BTU/hr ft}^2 \text{ }^\circ\text{F}$$

$$\text{or} \quad U = \frac{627,000 \text{ W}}{22 \text{ m}^2 \times 74.0^\circ\text{C}} = 385 \text{ W/m}^2 \text{ }^\circ\text{C}$$

Chart for Estimating Log Mean Temperature Difference



To use the chart above, line up ΔT_1 on the left scale with ΔT_2 on the right scale and read ΔT_{LM} from the middle scale. The values of ΔT_1 and ΔT_2 must be determined according to the rules for co-current and counter-current flow as described on page 4-8. The chart may be used with temperature in $^\circ\text{F}$ or $^\circ\text{C}$. To accommodate temperature values above the range of the chart, simply multiply all scales by a factor of 10.

Temperature Control Units

Traditionally, industrial temperature control was often accomplished by using high-pressure steam for heating or by circulating brine solutions for cooling. However, many modern pilot installations employ single-fluid closed-loop temperature control units (TCUs) because of the advantages they offer in terms of reliability, precise temperature control, wide operating range, low maintenance and no need to change out the fluid when switching from heating to cooling. A schematic of a typical single fluid TCU is shown at the bottom of the page. The major components are discussed in more detail below.

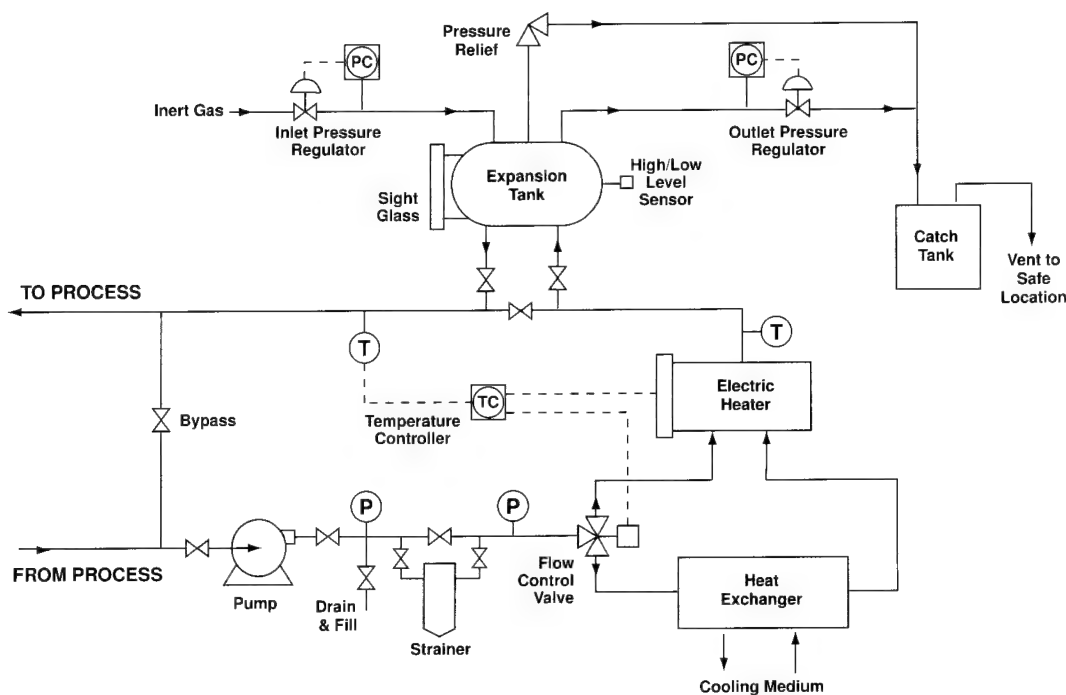
Heater – Most TCUs use electric elements for direct heating of the heat transfer fluid. Fluid velocity and turbulence must be sufficient to avoid excessive film temperatures that can damage the heater and shorten fluid life.

Pump and Piping – The pump must have sufficient capacity and output pressure to circulate the fluid at the required rate. At very low temperatures, for example, the high viscosity of some fluids may require a positive displacement pump to maintain good velocity. Many low-viscosity heat transfer fluids tend to leak, in which case, sealed magnetic drive pumps are preferred. Pump and piping materials, seals and gaskets must be compatible with the fluid over its full temperature range. A straining system (20 mesh or finer) should be included in the design to catch particulates, especially during start-ups, cleaning and maintenance. For very low viscosity heat transfer fluids, such as Syltherm or Therminol, gaskets should be class 300 flexitallic-type spiral wound, fittings should be brazed or welded, and packings for globe or gate valves should be spring-loaded graphite.

Expansion Tank – The expansion tank is usually located at the highest point in the system. It should be sufficiently large to allow for expansion and contraction of the fluid over its full temperature range while remaining between 25% and 75% full, and still supply the pump with uninterrupted flow. To prevent oxidation and absorption of moisture that can shorten fluid life, expansion tanks should not be vented to the atmosphere, but protected by a two-regulator inert gas blanketing system. The tank also should be fitted with a sight glass, a high-level alarm, a low-level cutoff safety switch and a pressure relief system.

Insulation – Use closed-cell type insulation to prevent saturation with HTF, which can create a potential fire hazard.

Schematic of a Typical Temperature Control Unit (TCU)



Safety Controls – Proper safety devices must be included to ensure safe system operation under all conditions. In addition to the expansion tank safety devices mentioned above, these should include: high temperature heater cutoff switch, heater low-flow cutoff, and safety relief valves vented to a safe location.

Process Control – Full microprocessor-based control is recommended so that the user can select any precise heating or cooling rate or temperature ramping profile. The ability to directly download and log temperature/time data from the unit will also prove very valuable in process optimization and scale-up.

System Selection – When selecting or designing a TCU, a number of important factors should be considered at the outset to ensure safe and satisfactory performance. The size and design will be significantly affected by the nature of the process (batch or continuous), the peak heating and cooling requirements, the characteristics of the heat transfer fluid, and the expected heat losses and pressure drop through the system. Most manufacturers and vendors of process TCU's (for example Budzar, Inc. and HEAT, Inc.) will use data provided by the user to ensure that the system meets all the necessary criteria. Manufacturers will generally provide a worksheet on which the customer can list all of the information required to properly size and design the unit. This will include data on the size, materials and construction details of the reactor vessel and associated piping, the characteristics of the batch fluid, the process requirements such as temperature range, peak heating or cooling rates and desired control options, the distance from the reactor to the temperature control equipment, the type of environment in which the unit will be situated (indoors, outdoors, or in a hazardous location), space or work environment limitations, budgetary constraints, etc. The manufacturer will provide you with the recommended design details, but completing the system design is an iterative, cooperative process. This is why it is important to work with experienced, reputable equipment suppliers. Many vendors will also offer services for installation, start-up and testing. An excellent source of further information on TCU design and practical advice is the TCU system design guides provided by Solutia and Paratherm Inc. [192, 236].

System Start-up and Operation

Start-up – Manufacturing debris such as welding slag, metal filings, cutting oils and other contaminants in new installations can cause damage to system components and promote fluid degradation. Therefore, make every effort to ensure that systems are completely clean and dry before introducing heat transfer fluid. Do not use water for pressure testing since water is difficult to remove, accelerates fluid degradation at elevated temperatures, and can cause corrosion. Water can also form potentially dangerous steam pockets at high temperatures and can freeze at low temperatures causing icing and fouling of heat transfer surfaces or damage from expansion. It is better to pressure test with an inert gas or a solvent that is easily removed.

Should water contamination occur, remove it by periodic low-point draining while operating the system. Removal of dissolved water is best accomplished by slowly ramping the system temperature and allowing the water to steam off through the expansion tank vent.

To prevent fluid oxidation, remove air by purging the system with an inert gas prior to charging the HTF. Charge using a small centrifugal pump connected to a system low point. Filling from the bottom in this way allows for natural venting and prevents pump cavitation. Upon start-up, filters should be checked frequently for any signs of system manufacturing debris or other contaminants.

Operation – The system should be brought to operating temperature slowly to avoid thermal shock of the system and to give any residual water a chance to steam off. Never operate outside of the recommended fluid temperature range.

Inspect the system daily by checking and recording pressure across filters or strainers, expansion tank pressure and level, and other operating indicators. Listen for unusual noises from the system, check for unusual smells, and periodically inspect a sample of the fluid for signs of water or anything else out of the ordinary. These practices can help detect problems before they escalate.

Shutdown – When shutting the system down, continue to circulate fluid with the heater off until the temperature in the system reaches a safe temperature (generally below about 50°C) before turning the system off. This will ensure that no local hot spots remain on heater surfaces that can overheat the fluid and accelerate degradation.

Tips on Using Heat Transfer Fluids in TCUs

The use of heat transfer fluids offers many advantages over steam heated systems, including better precision and uniformity of temperature control, lower maintenance, improved personnel safety and long service life. However, important precautions must be taken to ensure their successful use. Virtually all organic heat transfer fluids will oxidize on contact with air causing fluids to become acidic and sludgy resulting in solids formation, fouling, and diminished heat transfer capacity. Mechanical seal function can also be affected. For these reasons, air should be kept out of the system. This can be done by including an inert gas blanketing system on the expansion tank, and by purging the system with inert gas before charging. Air in the fluid can also be minimized by allowing cold fluids to equilibrate at room temperature prior to introducing them into the system.

Reactions between various heat transfer fluids at elevated temperatures are unpredictable. Therefore, never mix or combine heat transfer fluids in your system. Always ensure that the old fluid is completely flushed out of the system before replacing it with a different fluid (see below).

Overheating of the fluid at the heater surface can cause degradation and the buildup of catalytic products that can lead to more degradation and fouling. Many such problems can be prevented by good system design, periodic fluid testing and operating the system within the recommended limits for the fluid. Avoid exceeding the maximum recommended film or bulk temperature for the fluid in use.

Organic HTF systems are routinely operated at temperatures in excess of their flash and fire points. However, they must never be operated in excess of their autoignition temperatures, the point at which the fluid vapors will spontaneously ignite on contact with air even in the absence of an ignition source.

Fluids should be checked at least annually for contaminants and degradation, and more often if the fluid has been subjected to harsh conditions or extremely high temperatures for prolonged periods. Most fluid manufacturers will perform the analysis on their fluids for free. Contamination can also appear as reduced heat transfer rates, blockages of small diameter lines or fittings, and extended start-up times. Visually check a sample of the fluid for solids. Rust, pipe scale, dirt or other debris could indicate that the system needs to be flushed or even chemically cleaned. In the case of glycol-based fluids, degradation can slowly consume the corrosion inhibitors that are necessary to protect metal components. pH (and thus reserve alkalinity) is a good measure of how much corrosion protection remains.

Pressures are usually moderate in HTF systems, but there can still be slow leaking, or “weeping”, at pump seals, valve packings, and threaded fittings, especially for low-viscosity synthetic fluids. Follow the fluid manufacturer’s recommendations for gaskets and seals. Leaks can be particularly dangerous if insulation becomes saturated with fluid. Oxidation can then cause the buildup of temperatures in excess of the autoignition temperature, thereby causing a fire. It is important to locate and stop fluid system leaks promptly. Keep the system clean to help prevent fires and other mishaps.

Heat transfer systems are usually designed for a specific HTF, since the properties of the fluid will affect pump, heat exchanger, piping and vent sizes as well as other components. Therefore, one fluid is typically used for the life of the system. However, if it does become necessary to replace the fluid with another, it is very important to consider the effect of the new fluid properties on the operation of the process and on the sizing and rating of pressure relief systems, the expansion tank, seals and gaskets, etc.

Draining and flushing the system should be planned carefully. When draining the fluid from temperature control systems, warm the fluid first to reduce the viscosity and promote faster draining. Continue fluid circulation as long as possible during draining to keep any solids or debris suspended. Drain as much fluid as possible from all low points, since residual fluid can interfere with cleaning or can contaminate the fresh fluid charge. Compatibility issues between the new fluid and the old may require extensive flushing, cleaning and drying of the system. Sometimes, off-line filtration or chemical system cleaning may be necessary.

Fluid replacement and system restart should be undertaken with the advice and involvement of the new fluid manufacturer or supplier – contact them for more information. Finally, remember that all used HTFs must be stored, handled and disposed of in accordance with the appropriate environmental guidelines (see page 9-10).

Heat Transfer Fluid Selection

Selecting the best heat transfer fluid is critical for safety and efficiency, and involves many important considerations. Fluid stability and thermal characteristics will affect the selection of pumps, piping and heat exchangers and will determine the available temperature range and operating economy. Both liquid phase and vapor phase heat transfer media are used in the CPI. Vapor phase systems can deliver much more heat per pound of fluid, but their use is limited to applications between 180°C to 400°C. Since many liquid phase HTFs perform well at these and much wider temperature ranges, and because they offer greater simplicity of use, the discussion here is limited to liquid phase media.

The primary consideration is the required temperature range. Then the other factors of cost, thermodynamic properties, expected fluid life, operating pressure requirements, and environmental concerns are taken into account. The reputation and additional services offered by the fluid manufacturer also enter the decision. The thermal stability of the fluid should be considered carefully, based on the maximum expected operating temperature of the system. With prolonged heating, most fluids will break down into low-boiling compounds that are often vented off, and high-boiling compounds that can build up in concentration and affect fluid performance and ultimately shorten fluid life. These effects are accelerated when a fluid is heated beyond its recommended maximum operating temperature.

The major families of heat transfer fluids, besides water, are the inhibited glycols, silicone-based oils, mineral or paraffinic oils and the synthetic organic fluids. **Water** is cheap and plentiful but suffers from the obvious low temperature limitation of 0°C and extremely high vapor pressure at elevated temperatures (~1500 psi at 300°C). Water also often needs to be treated with corrosion inhibitors or other additives.

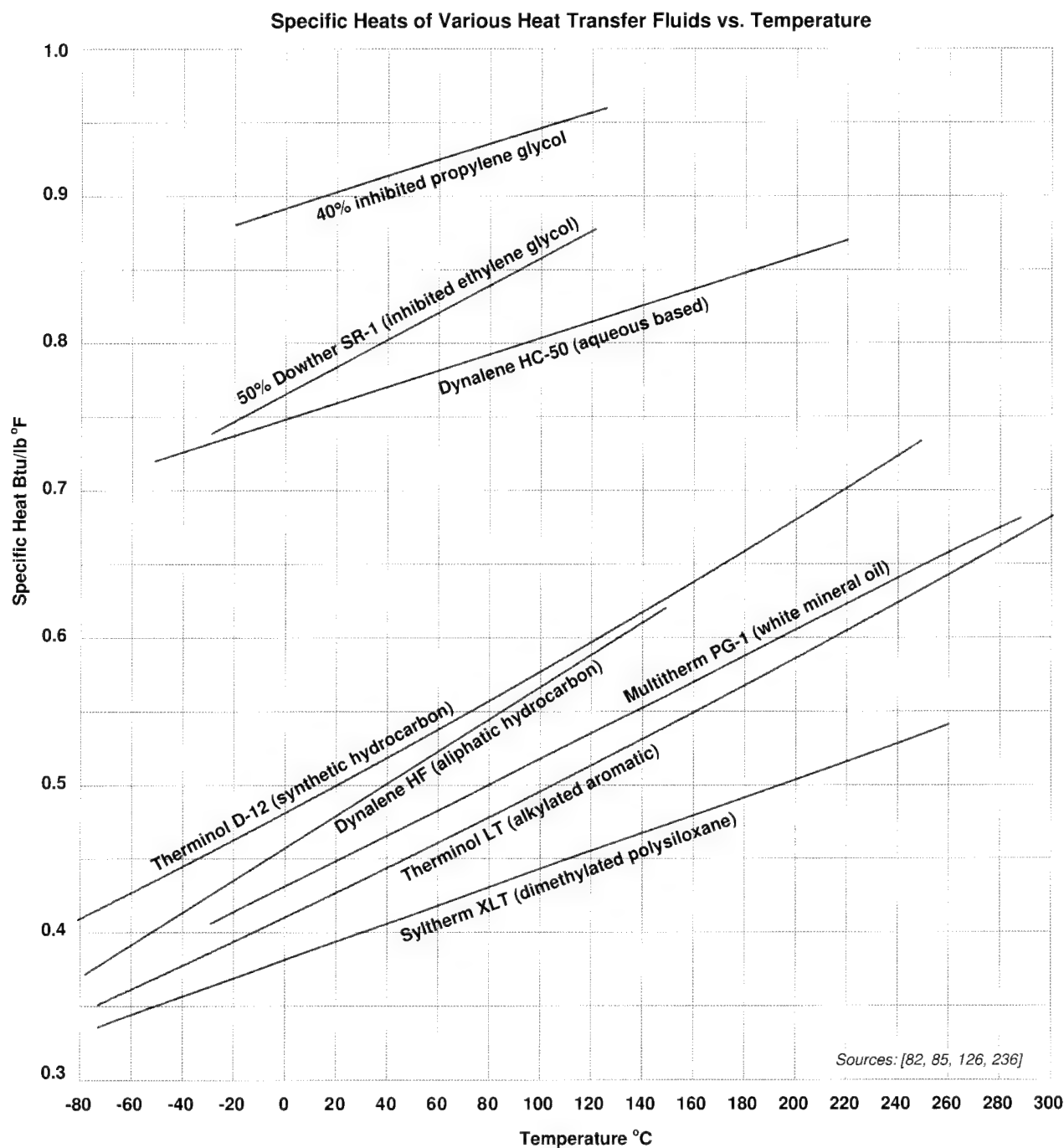
Calcium chloride or sodium chloride **brines** were once common in refrigeration, but without the addition of inhibitors, they are corrosive to many metals. They also suffer from decreasing specific heat with increasing concentration. Methanol, ethanol, acetone, glycerine, dichloromethane and others have also been used as freezing point depressants. The freezing point characteristics of some of these brines and traditional solutions are shown on page 4-24.

Glycol based fluids (ethylene or propylene glycol with inhibitors added) are probably the least expensive fluids available. They also have very high heat capacities and thermal conductivities. They are usually diluted with water for use and, depending on the mixture, can be pumped down to as low as -40°C, and can provide freeze and burst protection to as low as -50°C. More detailed information on the various glycol fluids is provided beginning on page 4-18. The **silicone oils** (polydimethyl siloxanes) may be more expensive initially, but are often economical choices because of their excellent thermal stability and extremely long fluid life (up to 10 years). They exhibit low odor and low toxicity and at elevated temperatures undergo an equilibrium-type thermal degradation as opposed to some other fluids that degrade irreversibly. However, they have very low viscosities and tend to leak if the system is not designed carefully. The **mineral or paraffinic oils** can also be economical choices in the 150 to 300°C range because of their long service life. However, they are usually too viscous to be pumped at low temperatures.

A whole range of **proprietary synthetic organic fluids** are also available. The chart on page 4-16 gives some indication of their number and their important properties. They include alkylated aromatics, aromatic ethers, alkylated diphenyls and hydrofluoroethers. These fluids may be quite costly but they offer the widest temperature ranges available and exhibit excellent thermal stability with long fluid life. Many are characterized by very low vapor pressure at elevated temperatures, and some are even recommended for use in open circulating baths. This should be discussed with the fluid supplier in detail prior to use in this type of application. Most of these types of fluids must be well-maintained in water-free and air-free systems to prevent irreversible thermal degradation. And while some exhibit quite low toxicity, some suffer from numerous regulatory or safety limitations.

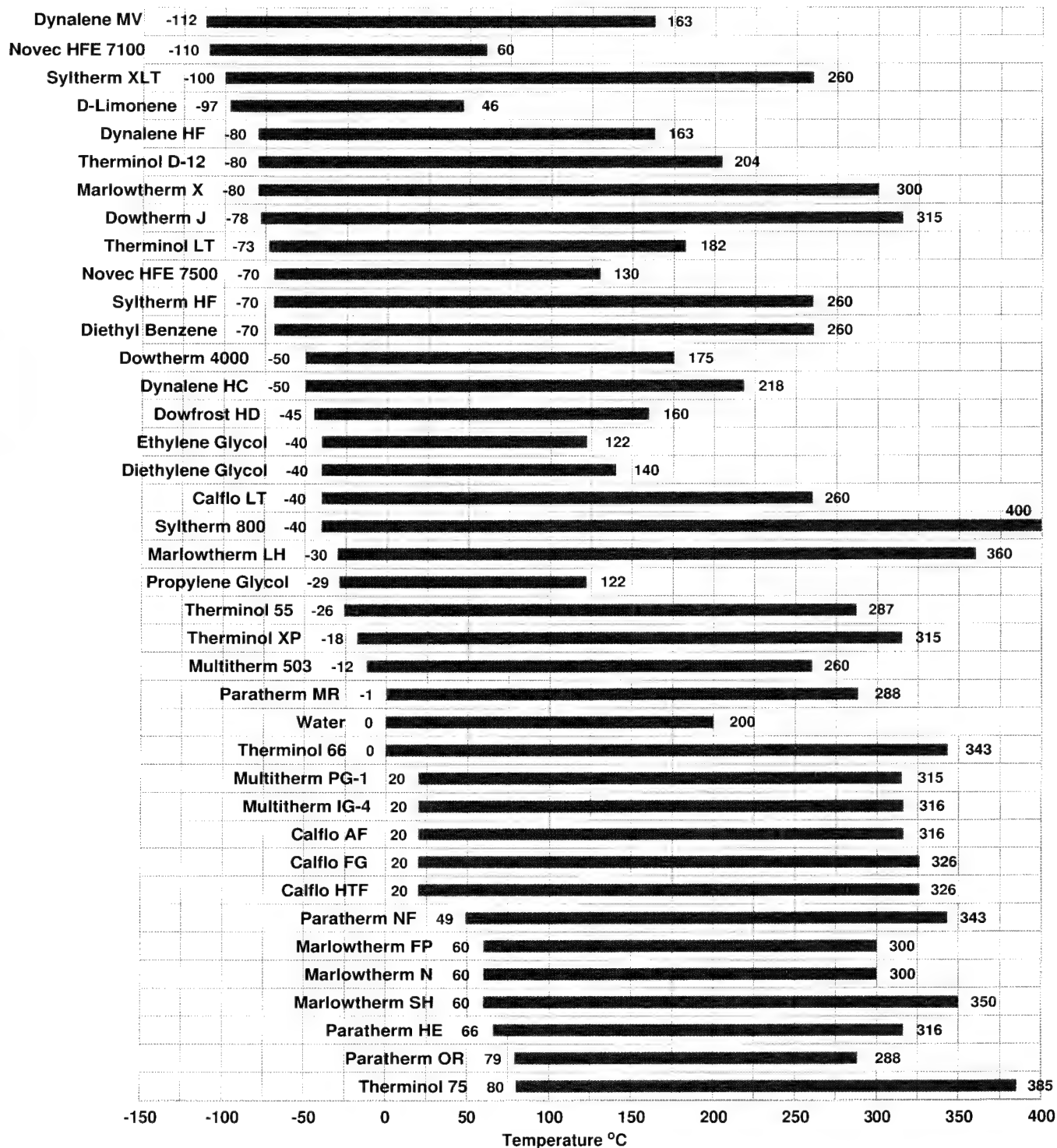
Most organic HTFs don't differ greatly in specific heat, density or thermal conductivity. However, since the viscosity of many increases dramatically at low temperatures, which in turn causes a drastic drop in heat transfer efficiency, the viscosity and efficiency at the low operating end should be well understood before committing to any product.

Ultimately, fluid choice should be a cooperative effort with input from prospective suppliers concerning your particular application. It is not worth trying to substitute a cheaper multipurpose oil for a good quality heat transfer fluid because the problems associated with fouling, corrosion and system damage make it more costly in the long run. It is usually more economical to go with proven high-quality products, especially in the demanding application of closed-loop TCUs.



The chart above illustrates that there is a wide range of specific heat (also called heat capacity) among heat transfer fluids. Since efficiency of heat transfer is directly related to how much sensible heat the fluid can absorb or hold, this is an important consideration in fluid selection. The glycols have the highest heat capacities, which makes them excellent choices for heat transfer media, but they suffer from other limitations as described on the previous page. At the other extreme, the dimethyl siloxane-based fluids have less than half the specific heat of the glycols, but they exhibit perhaps the widest useful temperature ranges of all the fluids. Each proprietary fluid included above is generally representative of its class; i.e., most synthetic hydrocarbons have specific heats roughly similar to that of Therminol D-12.

Useful Temperature Ranges and Properties of Commercial HTFs



The recommended temperature range information above is supplied in large part by the fluid manufacturers and does not reflect the possible need to operate the system under pressure to achieve elevated temperatures (see vapor pressure column for an indication of the vapor pressure in psia at the maximum recommended use temperature for

Manufacturer	Fluid Type	Approx. Viscosity cP	Approx. Density kg/L	Approx. Cp BTU/lb°F	Thermal Cond. BTU/hr ft²°F	VP at T _{max} psia	Atm. B. Pt. °C	Characteristics
Dynalene	Hydrocarbon blend	1.1	0.84	0.43	0.079	20	177	flammable
3M	Hydrofluoroether	0.58	1.51	0.43	0.040	28	61	prone to leaks, avoid TFE pipe dopes
Dow	Dimethyl polysiloxane	1.4	0.85	0.43	0.063	75	174	prone to leaks, flammable, FP=47°C
commercial	Citrus oil	3.5	0.84	0.48	0.069	0.2	176	corrosive, transport hazard
Dynalene	Aliphatic hydrocarbon	2	0.81	0.48	0.072	10	175	FDA food grade
Solutia	Synthetic hydrocarbon	12.0 (-50C)	0.76	0.52	0.062	57	192	low odor, FDA grade
Sasol	Substituted aromatic	0.71	0.803	0.43	0.073	158	180	low viscosity, distinctive odor
Dow	Alkylated aromatic	1	0.86	0.48	0.073	60	181	superior heat transfer to syltherm
Solutia	Alkylated aromatic	3.8 (-50C)	0.86	0.45	0.070	228	181	liquid or vapor phase
3M	Hydrofluoroethers	1.24	1.61	0.27	0.037		130	prone to leaks, avoid TFE pipe dopes
Dow	Dimethyl polysiloxane	2	0.88	0.44	0.063	50	210	high flashpoint
commercial	Alkylated aromatic		0.87	0.46	0.070	40	183	skin, eye irritant
Dow	Inhibited ethylene glycol	3	1.06	0.76	0.22	1	196	water soluble, good thermal char.
Dynalene	Aqueous-based	2	1.36	0.79	0.30	200	100	biodegradable, non-flammable
Dow	Inhibited propylene glycol	1	1.03	0.81	0.21	2	185	water soluble, good thermal char.
commercial	Ethylene glycol	17	1.117	0.83	0.15	1	197	water soluble, good thermal char.
commercial	Diethylene glycol	25	1.119	0.81	0.12	1	245	water soluble, good thermal char.
Petro Canada	Cracked hydrocarbon	6.3	0.82	0.48		4	323	odorless
Dow	Dimethyl polysiloxane	6.0	0.94	0.37	0.078	220	130	good thermal stability, prone to leaks
Sasol	Substituted aromatic	2.5	0.936	0.40	0.075	24	280	low viscosity, thermally stable
commercial	Propylene glycol	48	1.038	0.87	0.12	2	187	water soluble, good thermal char.
Solutia	Synthetic hydrocarbon	1250 (-25C)	0.87	0.47	0.073	7		
Solutia	White mineral oil	240 (0C)	0.88	0.45	0.071	6	358	FDA/USP approved
Multitherm	Paraffinic hydrocarbon	4	0.79	0.54	0.082	3	324	odorless
Paratherm	Paraffinic hydrocarbon	10.4	0.8	0.57	0.083	3	301	
		1	1	1.00	0.350	220	100	may require corrosion inhibitors
Solutia	Modified terphenyl	1320 (0C)	1.01	0.37	0.067	11	359	low vapor pressure
Multitherm	White mineral oil	18	0.88	0.46	0.076	5	349	FDA food grade
Multitherm	White mineral oil	38	0.87	0.48	0.077	2	411	thermally stable
Petro Canada	Cracked hydrocarbon	70	0.86	0.47	0.082	2	367	low VP, non-fouling, high lubricity
Petro Canada	Cracked hydrocarbon	31	0.86	0.46	0.079	2	382	USDA food grade
Petro Canada	Cracked hydrocarbon	31	0.86	0.47	0.082	2	392	odor-free, non-corrosive
Paratherm	Treated hydrocarbon	17.4	0.87	0.45	0.076	5	343	non-fouling, lower cost than glycols
Sasol	Hydrocarbon	40	0.797	0.48	0.076	8	340	thermally stable
Sasol	Alkylated aromatic	23	0.82	0.48	0.077	3	330	non-fouling, non-corrosive
Sasol	Dibenzyltoluene-based	20.7	0.987	0.39	0.074	3	390	thermally stable, non-corrosive
Paratherm	Paraffinic hydrocarbon	30.1	0.86	0.47	0.076	1	415	safe, high flash point
Paratherm	Treated natural hydrocarb.	52	0.87	0.47	0.088	1	333	additives prevent oxidation
Solutia	Synthetic aromatic	3.8 (100C)	1.04	0.40	0.083	31	343	soft solid at ambient temperature

each fluid). Also, the recommended minimum temperatures should be discussed in detail with suppliers, as thermal performance characteristics (specific heat, thermal conductivity and viscosity) can change drastically at low temperatures and severely affect heat transfer efficiency. Values are given for approximately 20°C unless otherwise indicated. Factors for converting the various fluid properties to metric units can be found in Chapter 11. Sources [67, 80, 81, 82, 85, 124, 126, 192, 196, 223, 236, 247].

Notes on the Use of Glycol HTFs

Inhibited ethylene or propylene glycol are economical, dependable heat transfer fluids if used within the appropriate limits. They have much higher heat capacities and thermal conductivities than synthetics, silicones and hydrocarbon fluids. They are perhaps the least expensive heat transfer fluid in common use and, when properly maintained, can have lifetimes in excess of 10 years. They can be used down to -40°C . The charts on the following pages provide information on the thermal and gravimetric properties of various mixtures of glycols.

Glycol products are typically diluted with water to provide a mixture with the desired temperature operating range. When making the dilutions, a 5°F safety margin should be provided to compensate for mixing and measuring errors. Over time, the water can evaporate off or additional water can be absorbed, thus changing the composition. Periodic sampling and analysis is recommended to ensure that the mixture stays within specifications and to ensure long fluid life. The formulas at the bottom of the page can be used to determine how much water or fresh glycol to add to a system to reestablish the desired mixture. Note that when water steams off at high temperatures, it can cause pump cavitation and dangerous venting. Thermal degradation is also a serious concern in glycol systems above 200°C . When overheated, glycols can form hard carbon deposits that foul heat transfer surfaces and cause corrosion.

Most commercially available glycols for heat transfer are sold with proprietary inhibitors added. Inhibitors are required to prevent corrosion, since reaction with oxygen at high temperatures produces acidic compounds that attack most common metals. The pH (and therefore the reserve alkalinity) is a good indication of the remaining level of corrosion protection. Glycols can usually be re-inhibited when necessary. Since most inhibitor packages are proprietary, it is necessary to get more specific information from the fluid supplier. Also, when diluting glycols to concentrations of less than 30 wt%, the inhibitor levels may need to be adjusted to ensure sufficient protection. One good reason to choose a proprietary inhibited glycol product is that the manufacturer knows the precise composition and can usually perform fluid testing and analysis for you.

Glycols do not have precise freezing points, but rather reach a "glass point" where viscosity becomes so high as to prohibit pumping. Likewise, aqueous glycol mixtures begin to form ice crystals and become slushy but can still be pumped until the temperature gets too low. This helps provide freeze and burst protection down to as low as -50°C . Approximately 60 wt% is the usable concentration with the lowest freezing point for most glycols.

Propylene glycol is considered nontoxic and is actually an approved food additive in some cases, whereas ethylene and diethylene glycol are much more toxic and not usable as antifreeze in potable water systems or domestic heating or cooling systems. Unfortunately, ethylene glycol has better thermal characteristics, but this must be weighed against the greater environmental impact. All used glycols, however, must be disposed of as hazardous waste.

Adjusting Glycol Concentration

The following procedure can be used to adjust the freezing point (concentration) of aqueous ethylene or propylene glycol solutions in heat transfer systems. V = total system volume, $[C_o]$ = the current or starting glycol concentration in *volume* %, $[C]$ = the final or desired glycol concentration in *volume* %.

To INCREASE concentration, A = volume to drain and replace with fresh glycol:

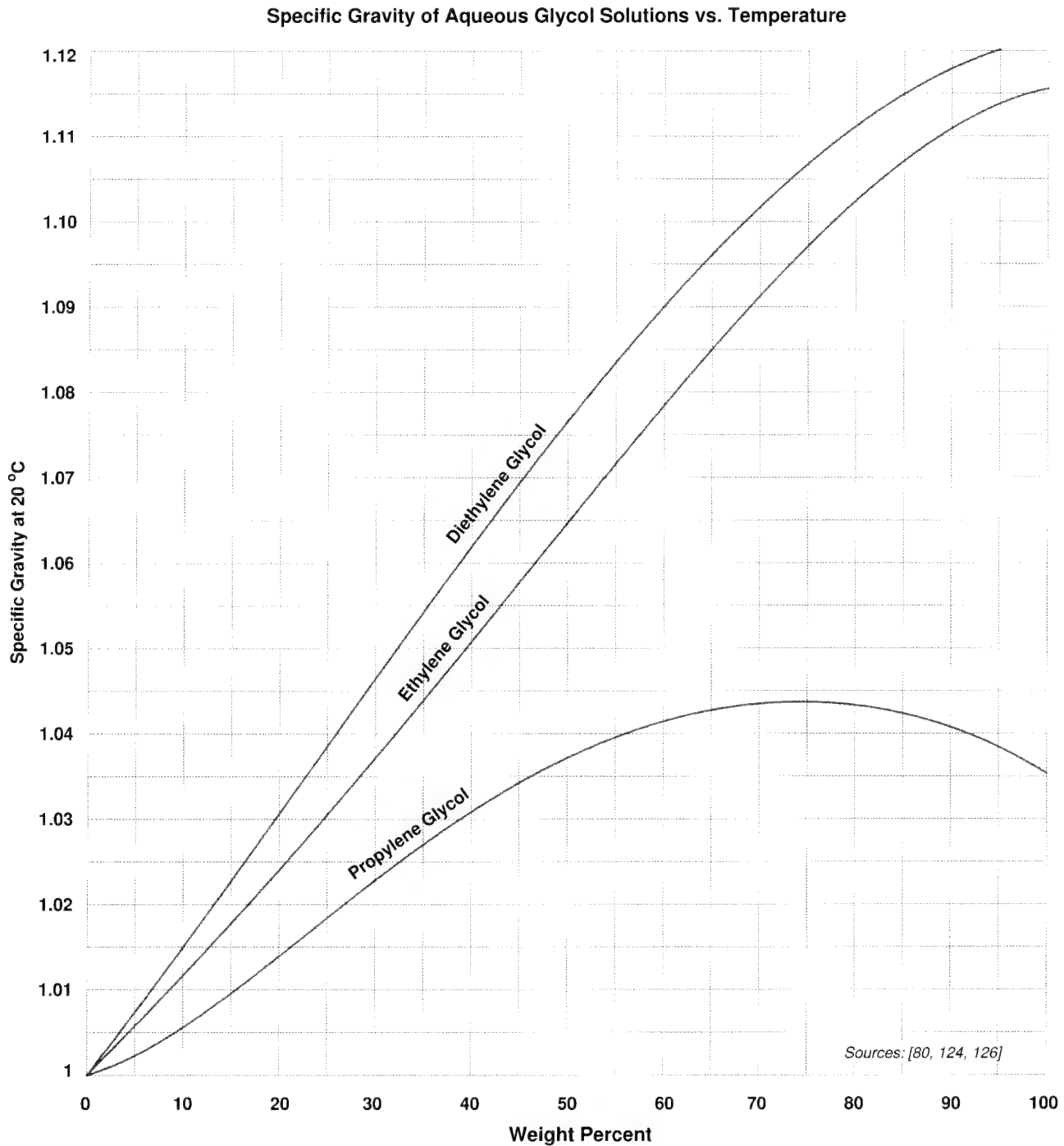
$$A = \frac{V \times ([C] - [C_o])}{100 - [C_o]}$$

To DECREASE concentration, A = volume to drain and replace with fresh water:

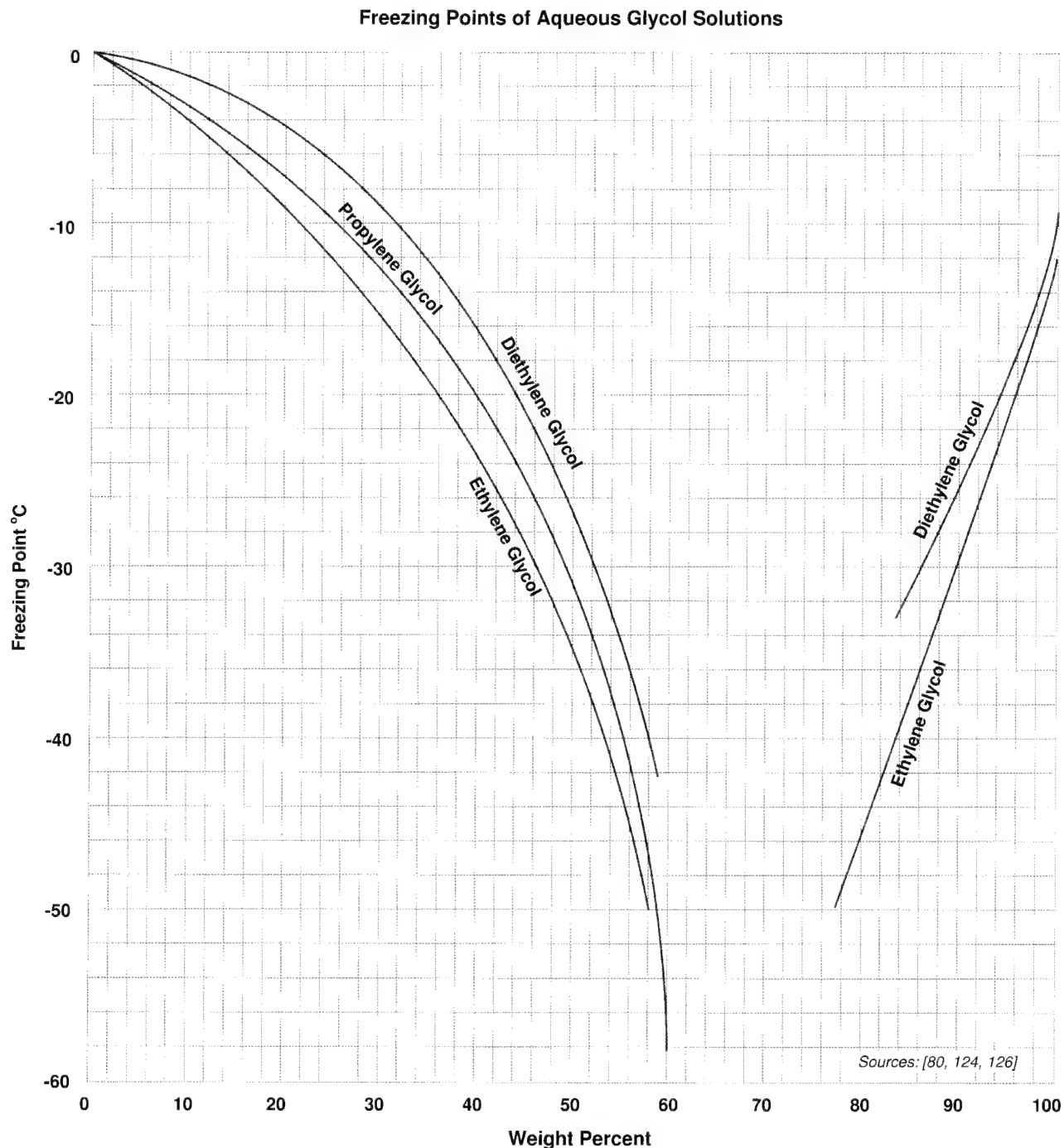
$$A = \frac{V \times ([C] - [C_o])}{[C_o]}$$

Wt % to Volume % conversion can be approximated by:

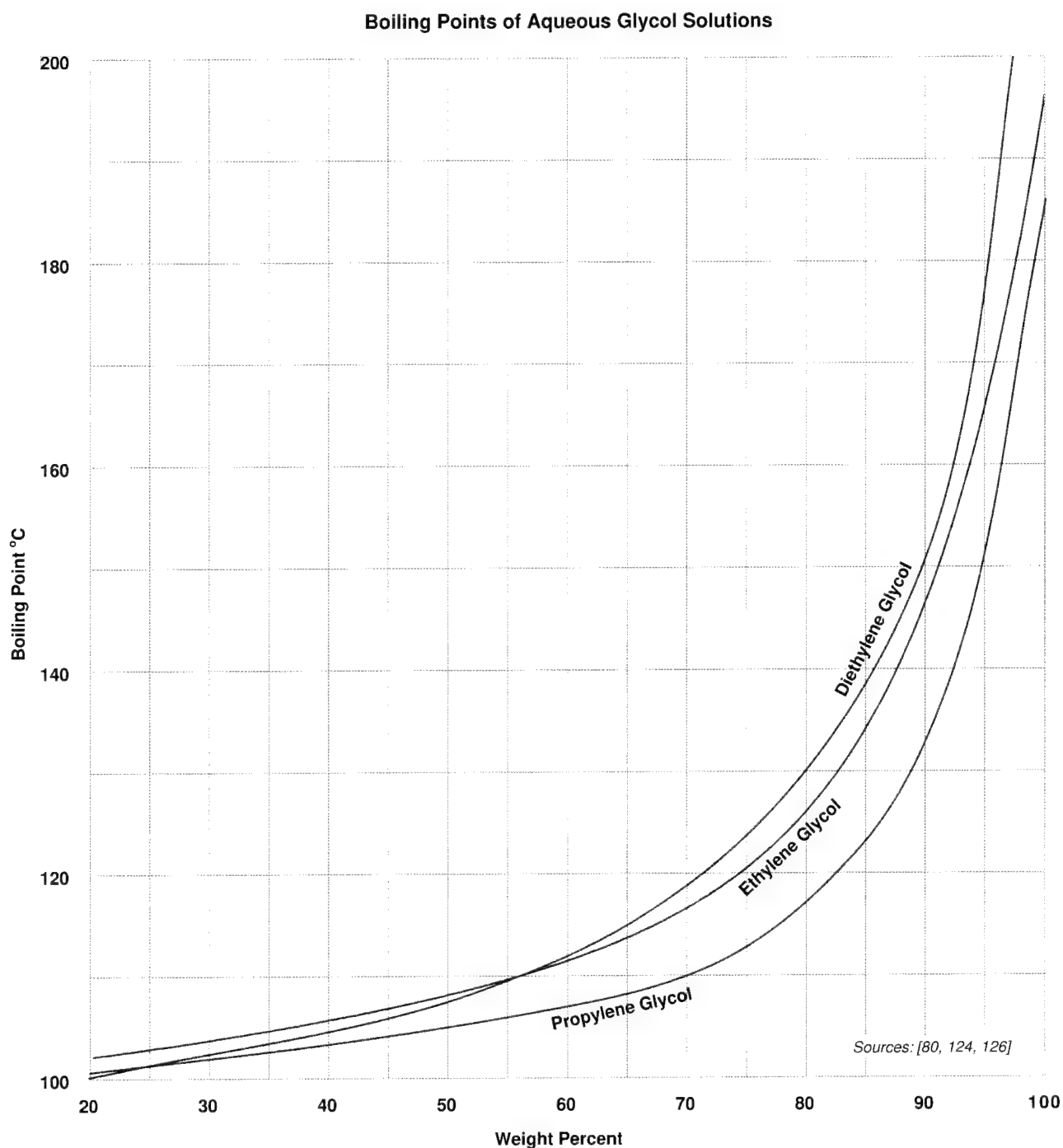
$$\begin{aligned}\text{Ethylene Glycol Volume \%} &= \text{weight \%} \times 0.931 \\ \text{Propylene Glycol Volume \%} &= \text{weight \%} \times 0.960\end{aligned}$$



The chart above shows the specific gravity at approximately 20°C of aqueous solutions of three common glycol heat transfer fluids as a function of weight percent. It can be used to correlate hydrometer measurements of glycol solutions to composition. Note that most glycol testing kits will only give correct results with ethylene glycol. It is better to use standard hydrometers, but make sure the sample is adjusted to the proper temperature. A rough correlation between weight percent and volume percent is given in the inset on the opposite page.

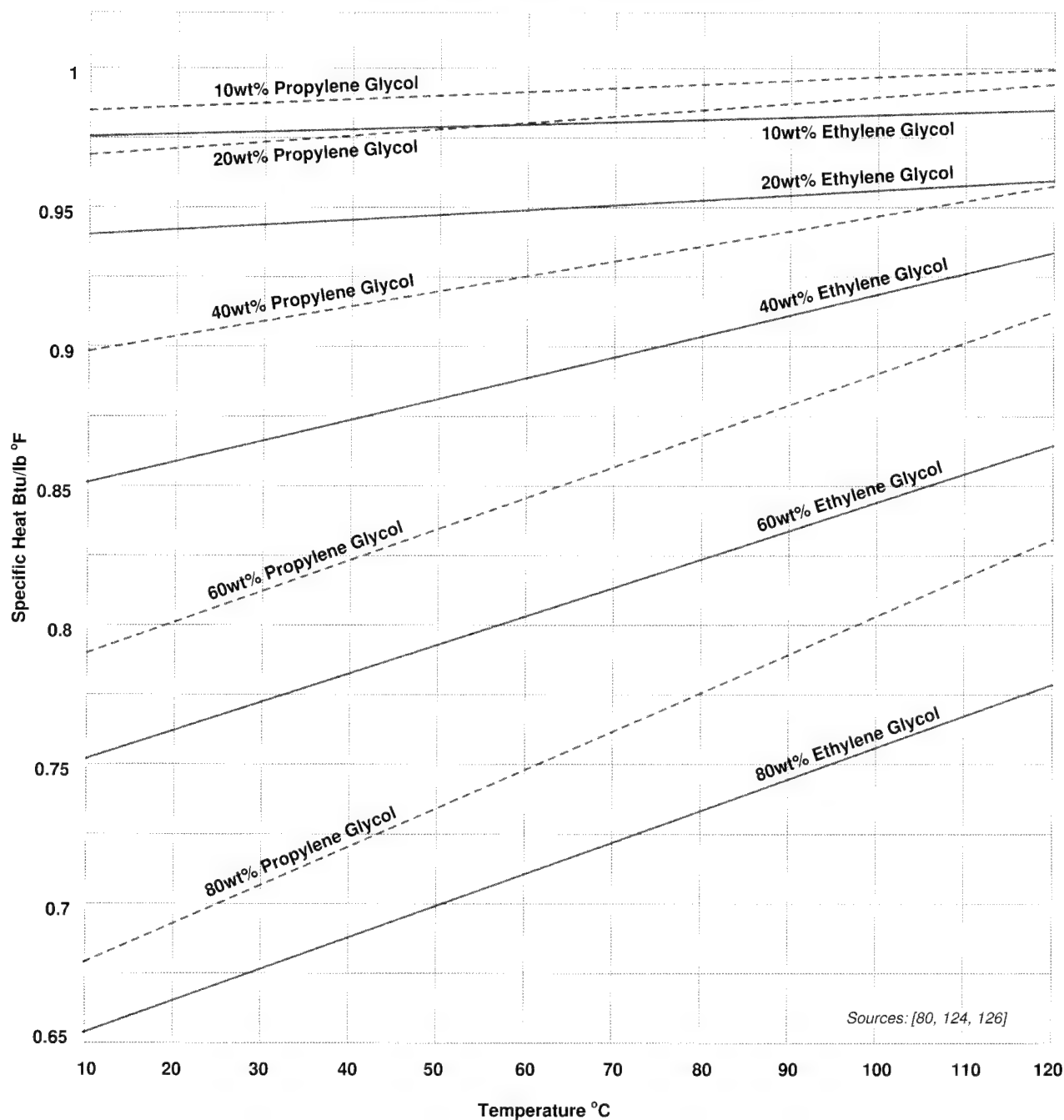


Glycols do not exhibit clear freezing points. Rather, at very low temperatures, the viscosity becomes so great that pumping is virtually impossible. This is called a “glass point”. Aqueous mixtures of glycols are characterized by another phenomenon. While the solution does not freeze per se, the water in the mixture begins to form ice crystals, creating a slushy mix that again is very difficult to pump. Note the discontinuities in the curves in the chart above. These represent glycol concentrations which, for the reasons described above, are unusable as heat transfer fluids at low temperatures. Thus for example, there would be no reason to prepare a 70% ethylene glycol solution, since its slush-point is considerably higher than that for 60% glycol.

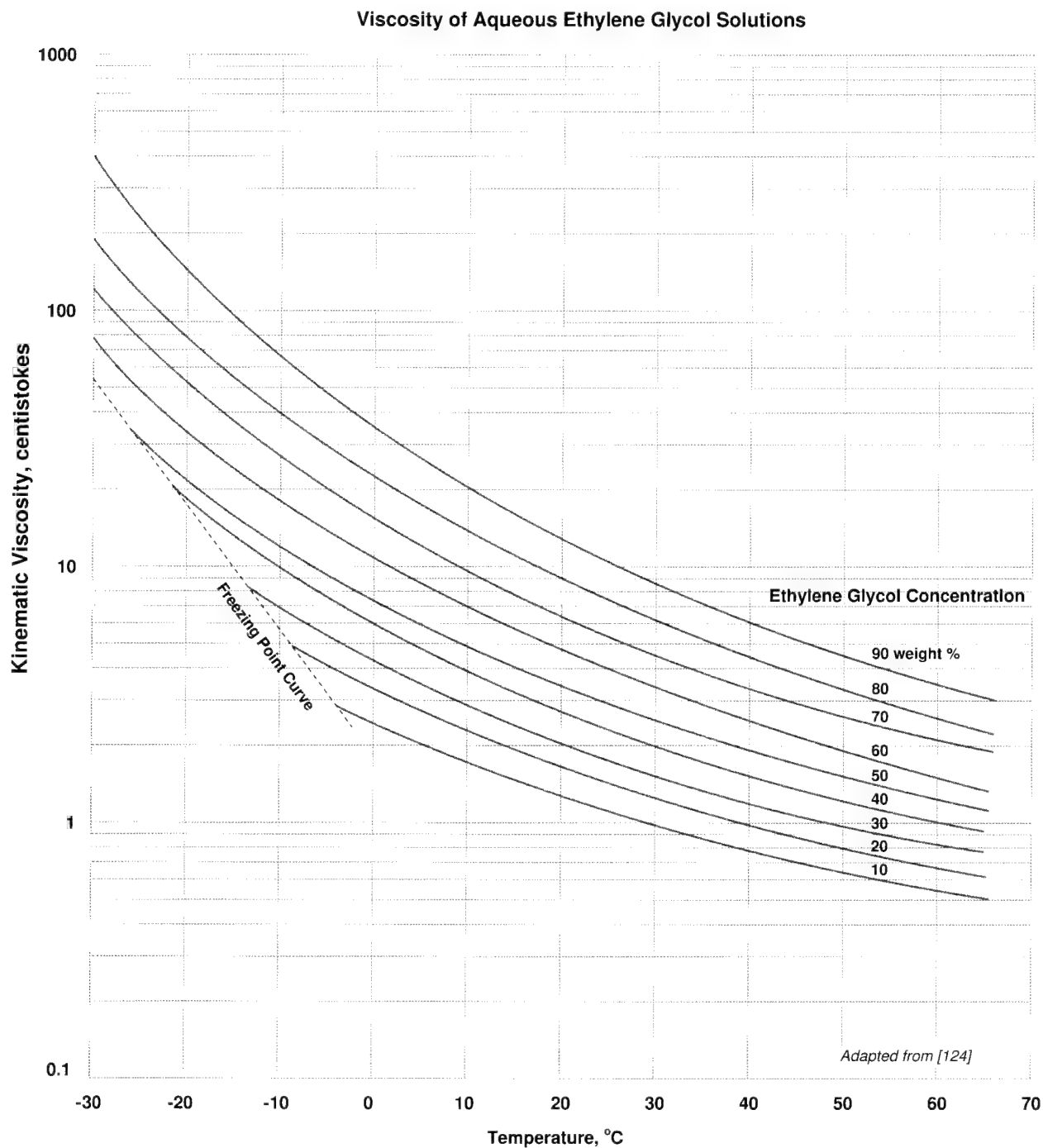


The chart above shows the boiling points at atmospheric pressure of various aqueous glycol solutions as a function of composition. These temperatures do not represent the maximum recommended use temperature, however. At very high temperatures, glycol solutions tend to oxidize, become acidic and sludgy. Proprietary inhibitor packages can extend the useful operating range, and so it is best to obtain details about your particular fluid from your supplier.

Specific Heat of Aqueous Glycol Solutions

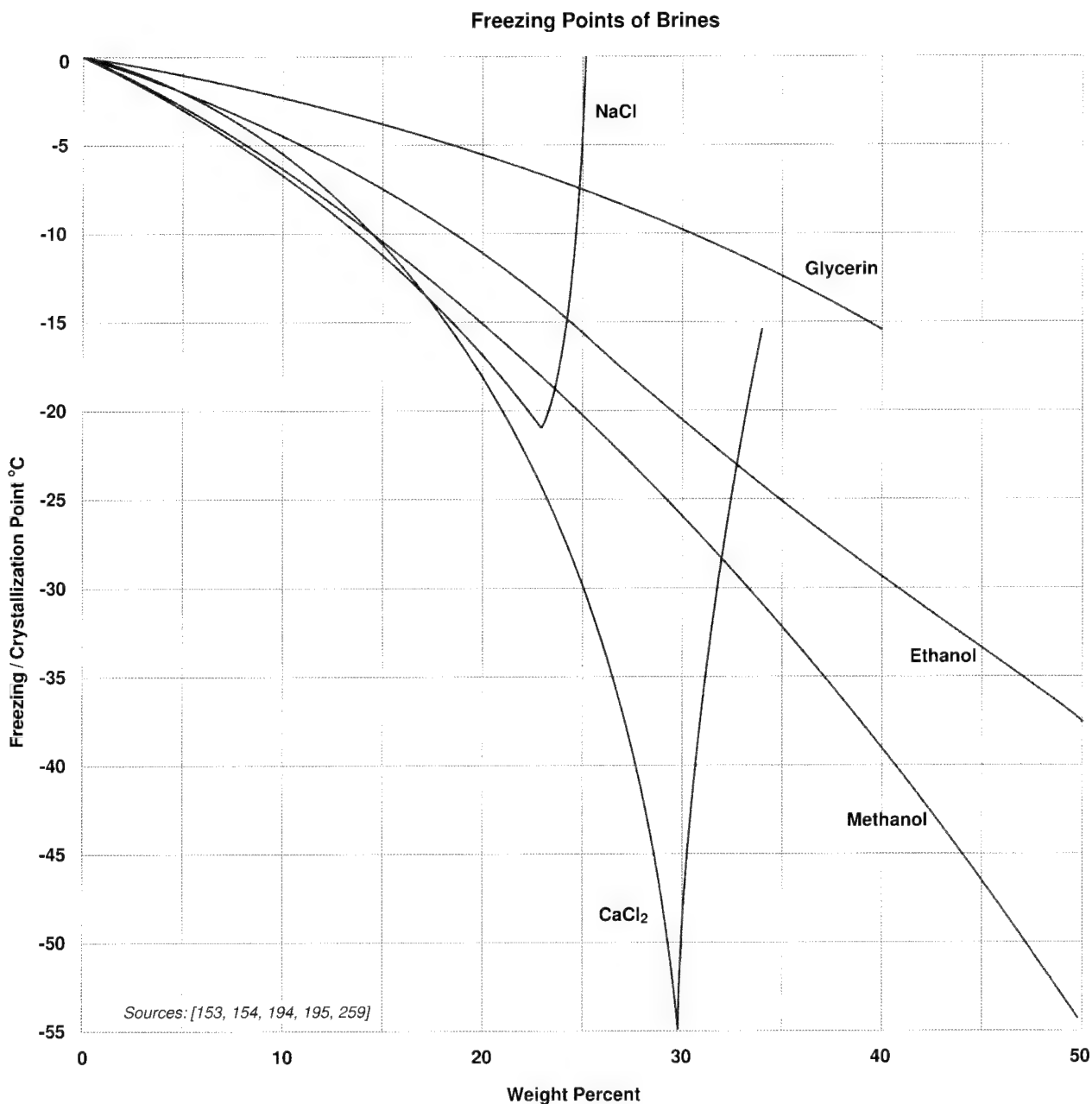


The chart above shows the approximate specific heat (or isobaric heat capacity, C_p) of aqueous ethylene glycol and propylene glycol solutions as a function of temperature. Compositions shown are in weight percent. A rough conversion from weight percent to volume percent is given in the inset on page 4-18.

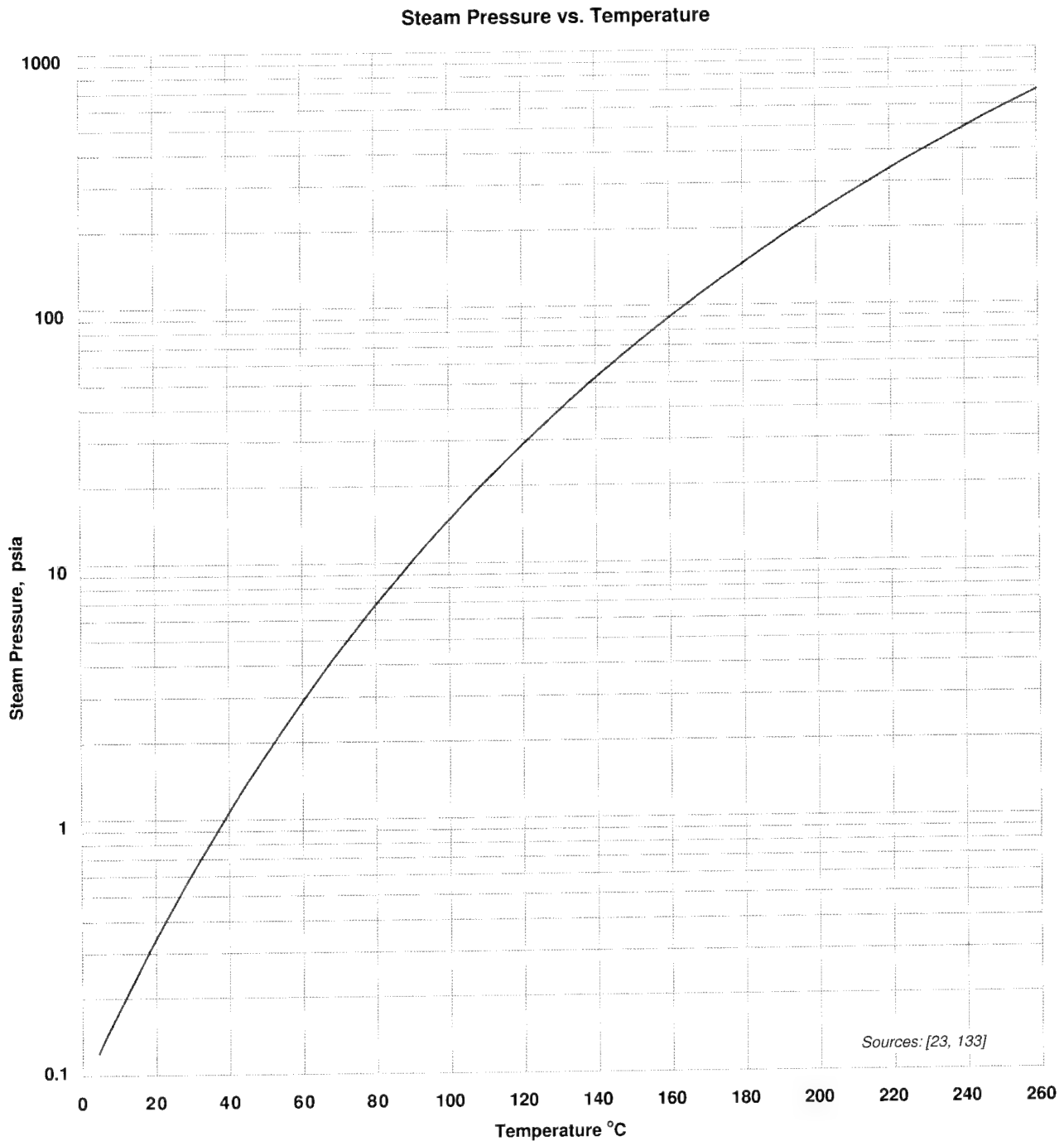


The chart above shows the kinematic viscosity in centistokes for various aqueous solutions of ethylene glycol. At low temperatures, concentrations of ethylene glycol above 60% are not recommended because the viscosity makes pumping difficult and heat transfer rates are severely diminished. Use the formula below to convert from kinematic viscosity to viscosity. Other conversion factors can be found in Chapter 11.

$$\text{Kinematic Viscosity (cSt)} = \frac{\text{Viscosity (cP)}}{\text{density}}$$



Brines or other solutions have traditionally been used in systems where low operating temperatures limit the use of water alone. NaCl and CaCl₂ brines exhibit minimum freezing points at certain critical concentrations (the eutectic). At lower concentrations, reducing the temperature causes ice crystals to form, producing a slush. At higher concentrations, reducing the temperature can cause salt to begin to crystallize from solution, eventually causing solidification. The major disadvantage of these brines is that they are corrosive and they are electrolytic, and therefore cannot be used in systems with junctions between dissimilar metals. Methanol and ethanol suffer from other disadvantages that include their flammability and high vapor pressures that can lead to evaporation and fluid loss. All of the above compounds have been largely replaced by the more thermally stable and noncorrosive glycols and the organic or synthetic heat transfer fluids. For a list of other mixtures used for cold temperature baths and their eutectic temperatures, see page 11-11.



The chart above shows the temperature of saturated steam as a function of pressure. Although steam is one of the most widely used heat transfer media, it suffers from a number of disadvantages, including the need for water filtration and treatment systems, costly corrosion inhibitors or other additives, and high operating pressures. In addition, steam generators may require full-time supervision by an approved boiler operator. Steam systems suffer from scale and mineral deposits that can foul heat exchange surfaces and require frequent maintenance of steam traps, condensate pumps, water analysis and treatment. Pressures in excess of 200 psia are required to achieve temperatures greater than 200°C.

Notes on Process Chillers

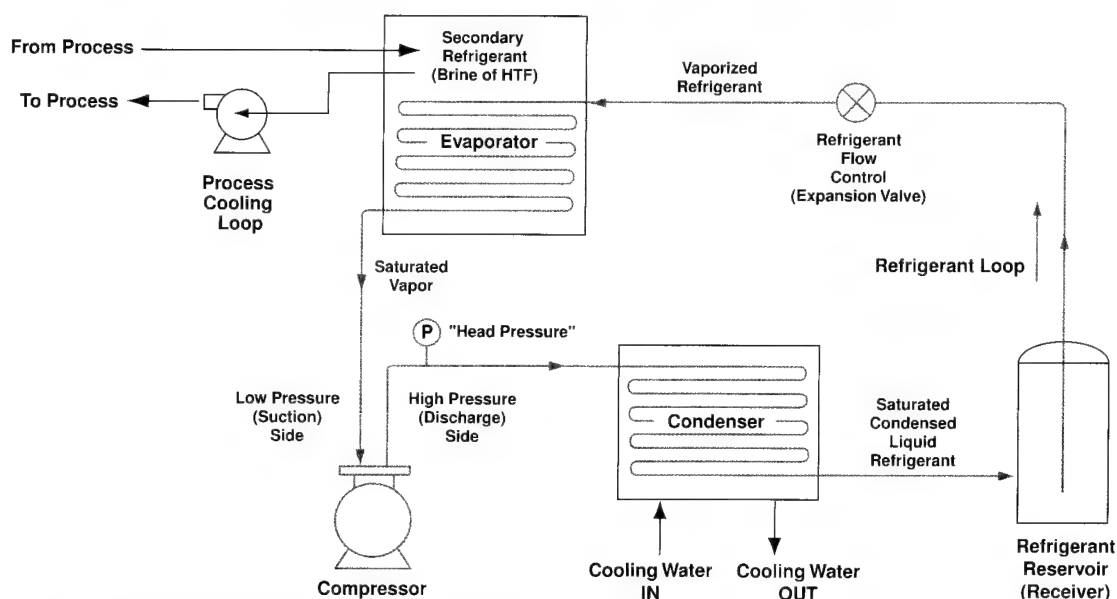
Process cooling is a fundamental operation in the chemical industry. Cold temperatures, down to as low as -80°C , are produced in chillers by the cyclic compression and evaporation of a specialized refrigerant in a system such as that shown in the diagram at the bottom of the page. The heart of the system is the compressor itself. There are many types of compressors, including reciprocating piston, rotary, screw, and scroll types (see page 7-16), and the choice of compressor is based on a number of factors, such as overall cooling capacity, temperature range and type of refrigerant. The term "hermetic" compressor refers to a system in which a compressor (usually piston-type) and motor assembly are sealed together in a vessel that is bolted or welded closed. They are used on small to medium sized units. A single-stage compressor is adequate for moderate sizes and temperatures, but multi-stage compressors may be required for very cold temperatures. For even colder temperatures, liquid nitrogen or other systems are often used. Systems that operate below 0°F (-18°C) are often referred to as *cryogenic* systems.

As shown in the diagram below, the compressed refrigerant is discharged from the high-pressure side of the compressor. It is passed through the condenser, which removes heat from the refrigerant causing it to condense to its liquid state. To dissipate the heat, the condenser may be either liquid-cooled, wherein cooling water or a glycol solution is circulated around the condenser coils, or air-cooled, in which case heat removal is accomplished by blowing air over the condenser coils. The condenser removes both *sensible heat* (reducing the temperature of the vapor to the condensation point) and *latent heat* (the enthalpy of vaporization) from the refrigerant. Increasing the temperature of the cooling water or air, or reducing its flowrate, reduces the overall efficiency of condensation and therefore reduces the ultimate cooling capacity of the system (see the chart of chiller capacities on page 4-28).

The liquefied refrigerant is allowed to expand (and thereby cool) through the flow control valve into the low pressure space of the evaporator. Heat is absorbed from the environment or secondary refrigerant (brine or HTF) during the expansion process. The vaporized refrigerant is drawn back into the suction side of the compressor where the cycle repeats itself. The cold heat transfer medium (secondary refrigerant) is circulated to the process via a separate process cooling loop. This approach is called indirect refrigeration since a secondary cooling medium is used to remove process heat.

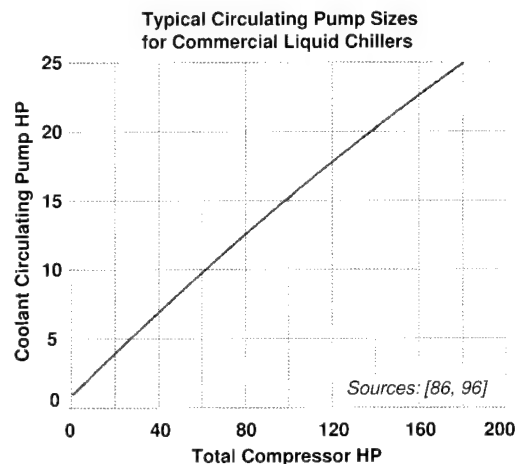
The circulating pump size for the process cooling loop depends on the chilling capacity. The chart at the top of the following page shows a rough correlation between pump size and compressor horsepower for typical chillers.

Components of a Basic Process Liquid Cooling System



Chiller Capacity – Cooling capacity is often referred to in terms of “tonnage” or “tons of refrigeration”. These are historical terms dating back to a time when ice was still widely used as a cooling medium. When one ton of ice melts, it absorbs 288,000 BTU of heat from the environment. Based on a melting time of one day, the heat is absorbed at a rate of 12,000 BTU per hour. Thus one ton of mechanical refrigeration = 12,000 BTU/hr of heat removal. The term can be confusing, since chiller capacity and heat removal rate are highly dependent on operating temperature.

When a manufacturer speaks of a “10-ton chiller” he may be slipping into a common industry usage where it is understood that the nominal capacity of the chiller is 10 tons (or 120,000 BTU/hr) at an operating temperature of 60°F. When operating at lower temperatures, the compressor has to work harder, and the capacity is very definitely lower. This is shown in the chart of chiller capacity on page 4-28. A more accurate way to refer to the size of a chiller is by stating the compressor motor horsepower value. For most compressors, the horsepower is roughly equal to the nominal tonnage.



Efficiency – Capacity is determined not only by the size of the compressor but also by the operating efficiency of the unit. This is in large part determined by the compression ratio, the difference in operating pressures between the suction and discharge side of the compressor. The lower the compression ratio, the lower the power consumption and the higher the volumetric efficiency. A system should therefore be designed to operate at the highest possible vaporization temperature and the lowest possible condensing temperature, thus maximizing efficiency. However, these values are ultimately determined by the physical characteristics of the refrigerant used, such as boiling point and condensing temperature.

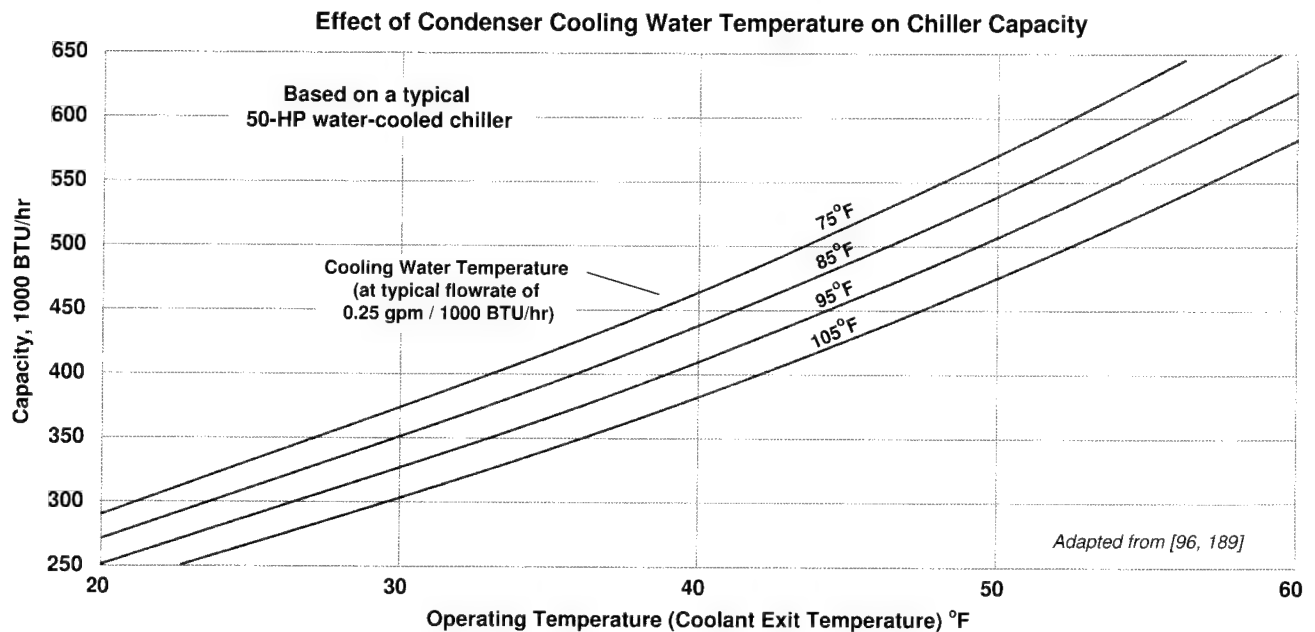
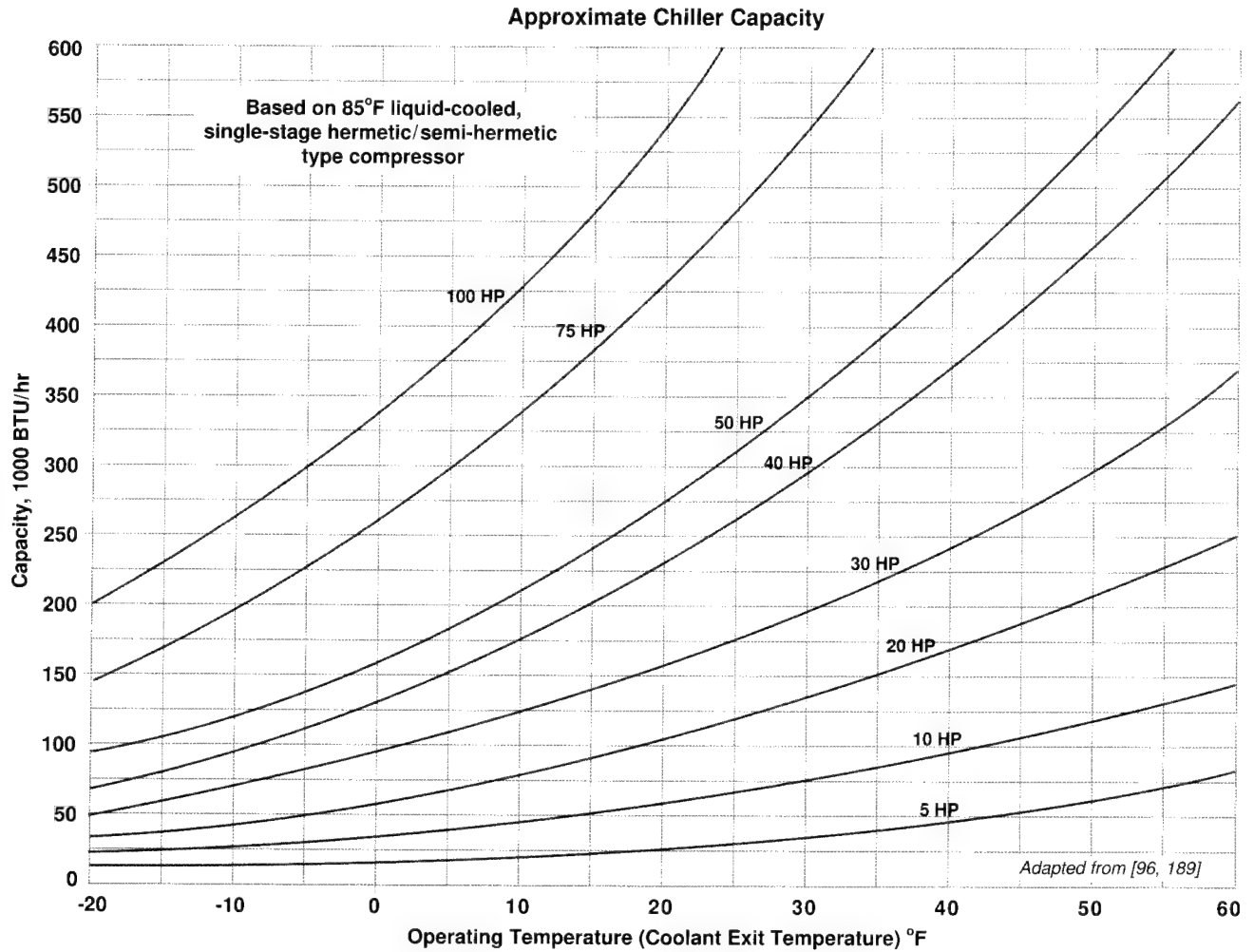
Refrigerants – The choice of refrigerant depends on several factors, such as its vapor-temperature behavior and efficiency, its environmental profile, toxicity, flammability, stability in the refrigeration circuit, and compatibility with materials of construction and lubricants. Ammonia is still used as a refrigerant in some very large applications, but the primary refrigerants used today are halocarbons. Because of their ozone-depleting and global warming potential, the fully halogenated CFCs (R-11, 12, 13, etc.) have been banned from use since 1996, and the partly halogenated HCFCs (R-21, 22, etc.) will be phased out and replaced with fully and partly fluorinated FC and HFC alternatives such as R-23, R-32 and R-41.

Sizing Chillers – When sizing chilling equipment, discuss your requirements in detail with a reputable refrigeration engineering firm (such as Carrier or Filtrine, Inc.). However, a rough estimate of the heat removal duty can be made by using the basic heat equation,

$$Q = MC_p \Delta T$$

where Q is the heat to be removed (in BTU), M is the mass of material to be cooled (lb), C_p is the specific heat of that material (Btu/lb °F), and ΔT is the desired temperature change (°F). The mass to be considered usually consists of not only the batch but also the mass of the vessel, the piping system, and the HTF itself, all of which must be cooled along with the batch. The *rate* of heat removal must be considered separately based on process needs. Remember that the highest rate of cooling usually occurs at the beginning of the cooling cycle (the peak duty), and sizing must take this into account. A safety factor of at least 10% should be added to your estimate.

Cooling Water and Air Requirements – A common rule for water cooled condensers states that approximately 3 gpm of 85°F (29°C) water is required per ton of capacity (or 0.25 gpm / 1000BTU/hr). If the cooling water is warmer than 85°F, the chiller capacity will be reduced, as shown in the figure on page 4-28. If warmer water must be used, either a higher flowrate or increased condenser heat transfer area can partly compensate. Obviously, for systems operating at temperatures near the freezing point of water, a glycol or other type of low-freezing solution must be used in place of plain water. For air-cooled condensers, the rule is 800 CFM of 95°F air per ton of capacity.



5 Electricity and Instrumentation

Contents

ELECTRIC POWER

Notes on Electrical Safety	5-2
Electric Power Basics	5-3
Wire Data	5-4
Standard Resistor Color Code	5-5
Tips on Making Electrical Connections	5-5
Receptacle Data	5-6

ELECTRICAL PROTECTION

Electrical Enclosure Data	5-7
Hazardous Location Classifications for Electrical Equipment	5-8
Intrinsically Safe Equipment	5-10

MOTORS

Electric Motors	5-11
Motor Nameplate Data	5-11
Typical Current Draw of Induction Motors	5-12
Common Types of Motor Enclosures	5-13
Electric Motor Troubleshooting Guide	5-14
Standard NEMA Motor Frame Sizes	5-14

INSTRUMENTATION AND CONTROL

Temperature Measurement	5-15
The 4-20 mA Transmitter	5-17
Pressure Measurement	5-18
Flow Measurement	5-20
Level Measurement	5-22
pH Measurement and Control	5-23
Notes on Process Control	5-25
Understanding PID Controllers	5-26

Notes on Electrical Safety

The hazards associated with electricity are numerous, ranging from the danger of electric shock to the ignition of flammable vapors or other materials from sparks and resistive heat emitted by electrical equipment. Installation and use of electrical equipment in pilot plants and other hazardous locations involves extra precautions because the energy levels and potential dangers are multiplied. An awareness of the potential hazards and some basic precautions can go a long way in preventing mishaps. Common sense dictates observing the precautions listed below.

General – Whenever possible, use equipment with three-prong grounded plugs. Never use equipment with frayed wires, broken insulation or missing cover plates. Avoid the use of excessively long extension cords or those smaller than the size required by the equipment. If a cord feels warm, disconnect it immediately. Avoid connecting too many pieces of equipment to one outlet, which can cause overheating or blow circuit breakers. Do not bury wires under carpeting or place other objects on them. Do not string cords near sinks or water sources or over metal pipes or ducts. Keep all equipment away from water unless it is properly rated and do not handle electrical equipment with wet or damp hands or while standing on a wet surface.

Receptacles – Electrical outlets should be protected by ground fault interrupts (GFI) that disconnect power if there is a short circuit to ground. Wiring polarity and proper grounding of outlets should be checked periodically by a qualified technician or electrician. Always observe the correct use of polarized outlets (see page 5-6). Wiring the receptacle incorrectly or plugging in an appliance with the plug reversed can make the entire outside surface of the unit “hot”. A voltage sensor is an inexpensive and extremely useful device that can be used to test for this.

Grounding – All electrical equipment must be properly grounded, so that if the “hot” lead accidentally contacts the case, the current is diverted to ground without endangering the user. The size of the grounding wire should be based on the voltage and the size of the overcurrent circuit protector. Secure grounding should be tested periodically by using a meter to check for continuity between all external metal surfaces and a known established ground point.

Circuit Breakers – Circuit breakers and fuses should be properly labeled and all personnel should be familiar with their location and use. If a breaker trips or a fuse blows, ascertain the cause and correct it before attempting to use the equipment again. Always replace blown equipment fuses with fuses of identical current and voltage rating.

Flammables – Do not use electrical equipment or appliances in the vicinity of flammables, unless you know that they are explosion-proof or specifically designed to prevent the emission of sparks from on/off switches and relays. Do not use conventional heating plates that are not spark-proof. Ovens should have adequate ventilation to prevent the build-up of flammable vapors. Refrigerators and freezers should have externally located control switches to keep sparks away from the interior. Do not block equipment vents or position too close a wall or in cabinets that could block ventilation.

Enclosures – Use only electrical enclosures that are properly rated for the location in which they will be used. National Electrical Manufacturer’s Association (NEMA) enclosure types and their applications are listed on page 5-7. Wiring must comply with all local codes, the National Electric Code (NEC) and UL specifications, as must all cable entry and conduit systems. A certified electrician should carry out all electrical equipment and installation work.

Hazardous Locations – Observe the classification of hazardous environments for the proper selection of electrical equipment, wiring and enclosures. See pages 5-8 through 5-12 for more information on hazardous locations and the selection and use of explosion-proof motors and other equipment. If intrinsically safe appliances will be used, they must be installed in strict compliance with the standards established by the NEC and other pertinent organizations. Good communication with equipment vendors is critical to selecting the appropriate equipment for your situation. Safety can only be assured by a close working relationship between the manufacturer, installer and end user.

Maintenance – When working on electrical equipment always make sure the power is off. It is wise to establish and adhere to a consistent Lockout/Tagout policy that complies with OSHA regulations to prevent serious injury, not only for electrical switches, but for compressed air, gas or any other utilities that store energy that may be used by the piece of equipment. Remove any metallic or other conductive jewelry in case it contacts a live circuit. Use only nonconductive (wood or fiberglass) ladders. Repairs or service of electrical equipment should be performed only by qualified personnel, wearing the appropriate protective equipment, including insulated gloves rated for the types of voltages involved.

Electric Power Basics

In the U.S., alternating current (AC) electricity is normally used in voltages of 120, 208, 240, 277 and 480V. These are considered *low voltage* levels, whereas anything above 600V is considered *high voltage*. Low voltage applications are by far the most widely used for small industrial equipment.

Typically, power is delivered to a building as 480VAC. It is then treated by a step-down transformer to produce lower voltage supplies. Depending on the wiring configuration of the transformer (commonly a “Y” connection or a “delta” connection; see diagram below) and which “legs” are tapped, the user can be provided with 120V for small appliances and single-phase motors, 208V or 240V for 3-phase motors, 277V, which is usually used for wide-scale industrial lighting, and 480V for larger 3-phase equipment such as 50HP or larger motors to drive compressors and processing equipment. **Single phase** means that the device is connected to only one line leg, a neutral and a ground. In **three-phase** equipment, the unit is actually connected to three sets of line legs, each of which supplies 1/3 of the power, and sometimes a neutral line. The cycles of the three voltages are out of phase with each other by 120°. Three-phase power offers important advantages for electric motors one of which is constant rather than pulsating torque (see page 5-11).

The reason for using different voltages has to do with the fundamental relationship between voltage, current and power. High voltages can deliver more power with less current draw. Thus, for a 50 HP compressor motor, 480V is often used so that the required power can be delivered with less current drawn than if 208V were used. This allows the use of smaller wires, since wire size must be matched to current draw to prevent overheating (see wire data on page 5-4).

Most equipment can accommodate slight variation in voltage, but significant deviation from the labeled rating can result in poor performance, overheating and premature failure. It is important to know the exact voltage requirements before installing any equipment. In some cases, a 110V or 220V label on a device may be a nominal rating and the equipment may operate correctly on 120V or 208V. In other cases, equipment labeled 230V may require exactly 230V, in which case, an additional transformer may need to be installed. Always consult the manufacturer when in doubt.

Because of the complexity of the NEC guidelines for various power levels, wire classes and insulation types, conduit and junction box capacities, specific overcurrent protection requirements, and the inherent risk of injury and damage and legal ramifications, electrical installation and maintenance work should be performed only by a licensed electrician.

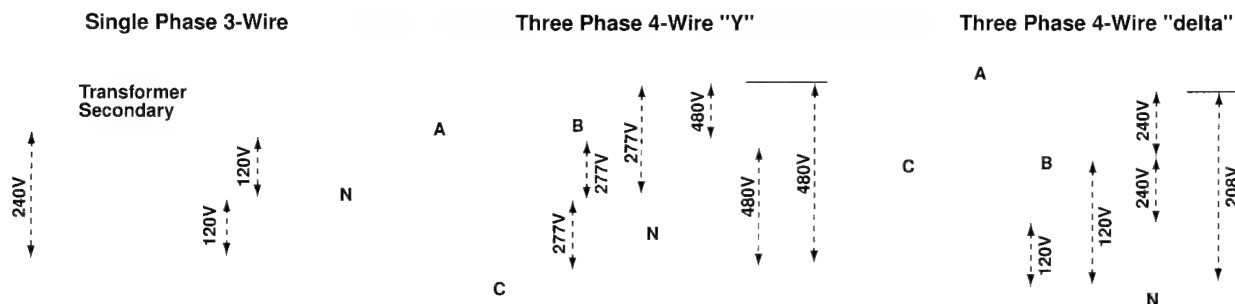
Determining Current Draw – It is often necessary to know the amount of current a piece of equipment draws. Some useful relationships between power, current and voltage are given below. By way of example, the current drawn by a 120V, 1000-watt light bulb is easily calculated as 8.3 Amps. Likewise, current drawn by a 1 HP 120V single phase motor is 6.2 Amps (this calculation ignores efficiency, which in small single-phase motors can be 50% or less). For more accurate estimates of motor draw, see the table on page 5-12.

$$\text{Current (Amps)} = \frac{\text{Watts}}{\text{Voltage}}$$

$$\text{Current (Amps)} = \frac{\text{HP} \times 746}{\text{Voltage}}$$

$$\text{Current (Amps)} = \sqrt{\text{watts} / \text{ohms resistance}}$$

Typical Step-down Transformer Wiring Configurations



Source: [173]

Wire Data

Wire and cable must be selected for each application according to NEC guidelines for wire size, material, insulation class, location approvals, etc. The table below shows the characteristics of some common copper wire sizes. The NEC wire type codes (such as TW, UF, RHH) indicate the insulation material, the full load temperature rating, wet/dry service and so on. There are many types and classes of wire. Selection must be made by qualified personnel.

Copper Wire Characteristics

Wire Gauge (AWG)*	Wire Diam. (inches)	Resistance $\Omega/1000$ ft (77°F)	Rated Current Capacity (Amps) at 86°F for 3-Conductor Copper Wire**			
			140°F rated (types UF, TW)	167°F rated (types RH, RHW, THW, THWN and others)	194°F rated (types XHH, TA, TBS, SA, RHH, THHN, others)	Lamp & Extension Cord (types SP, SPT, S, SJ, SV, ST, others)
0000	0.460	0.050	195	230	260	-
000	0.410	0.063	165	200	225	-
00	0.365	0.079	145	175	195	-
0	0.325	0.100	125	150	170	-
1	0.289	0.126	110	130	150	-
2	0.258	0.159	95	115	130	-
3	0.229	0.201	85	100	110	-
4	0.204	0.253	70	85	95	-
6	0.162	0.403	55	65	75	-
8	0.128	0.641	40	50	55	-
10	0.102	1.020	30	35	40	25
12	0.081	1.620	25	25	30	20
14	0.064	2.580	20	20	25	15
16	0.051	4.090	-	-	18	10
18	0.040	6.510	-	-	14	7

*AWG = American Wire Gauge **For ambient temperatures above 86°F, capacity must be de-rated. See [184].

Sources [8, 107, 173, 184]

Voltage Drop – The voltage drop in wire for a given application should be kept below 2% of the full line voltage if at all possible. The voltage drop in wire is proportional to the wire's resistance through Ohms law:

$$\text{Voltage drop} = \text{Current (Amps)} \times \text{Resistance (Ohms)}$$

This equation, together with the resistance data in the table, can be used to calculate the expected voltage drop. For example, for a 120V device drawing 5 amps, fed by 100 ft of #12 copper 2-conductor wire, the voltage drop will be:

$$5 \text{ Amps} \times 120 \text{ ft} \times 2 \times \frac{1.62 \text{ Ohms}}{1000 \text{ ft}} = 1.9 \text{ Volts}$$

This is 1.6% of the total line voltage of 120V and so the wire is appropriately sized for this duty. This relationship can be rearranged to determine the maximum allowable length of a given wire:

$$\text{Max. length (ft)} = \frac{\text{Allowable Voltage Drop} \times 500}{\text{Amps} \times \text{Resistance (Ohms/1000 ft)}}$$

Note that increasing ambient temperature above 77°C requires that the wire be de-rated according to NEC schedules (a 20°F rise in ambient temperature reduces current capacity by about 25%, but the exact value depends on wire size. Also note that the resistance values in the table are for copper wire only. Aluminum wire is typically rated for less current.

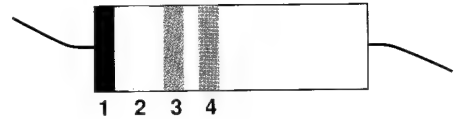
Typical 4-Wire Color Codes	
BLACK "Hot" or live lead	GREEN (or gr-yellow) "Ground"
WHITE "Neutral"	RED "Traveller" wire for 3-way switch

Some Common Wire Type Codes					
T (common) dry, < 60°C	S appliance, stranded	SPT lamp, plastic ins.	FEP FEP insul. T < 90°C	X... x-linked polymer ins.	...B outer braided
...H high temp. to 75°C	...HH high temp. to 90°C	...J medium service	...N extrud. nylon cover, tough	...V light service	...W wet use

Standard Resistor Color Code

The color code below is used to indicate the resistance in ohms of axial lead resistors. By way of example, if the resistor has bands of brown-red-orange, its resistance is 12,000Ω, with a tolerance of 20%.

Color	1st digit	2nd digit	3rd digit	4th digit (tolerance)
Black	0	0	1	-
Brown	1	1	10	1%
Red	2	2	100.00	2%
Orange	3	3	1,000.00	3%
Yellow	4	4	10,000.00	4%
Green	5	5	100,000.00	-
Blue	6	6	1,000,000.00	-
Violet	7	7	10,000,000.00	-
Grey	8	8	100,000,000.00	-
White	9	9	1,000,000,000.00	-
Gold	-	-	0.1	5%
Silver	-	-	0.01	10%
(none)	-	-	-	20%

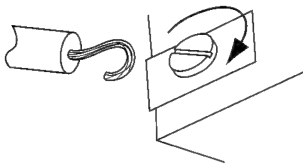
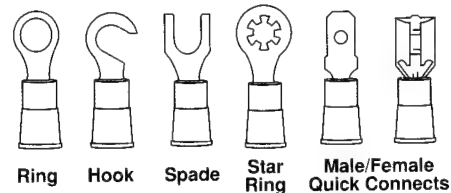


Adapted from [107]

Tips on Making Electrical Connections

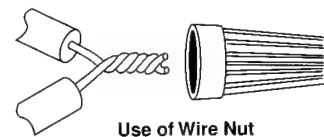
Many types of wire connections are approved for use in various situations. Assess the application carefully to ensure that the method is appropriate. Remember that vibration, temperature changes and the like can loosen some connections. Always use high-quality components from a reputable supplier. A few of the more common connection types, with some pointers for their use, are described below.

Solderless connectors are useful in a wide variety of applications, but they must be properly selected based on wire size, screw size and purpose. Ring and star ring types are best for permanent installations but hook and spade types ease assembly. Do not try to use pliers to crimp; always use a proper terminal crimping tool.

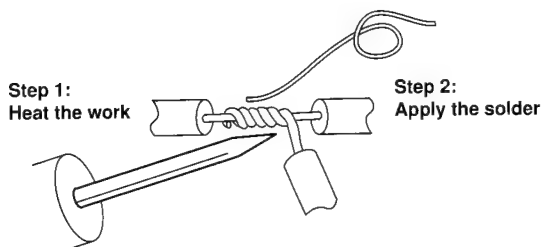


When screwing wires onto terminals without connectors, strip insulation and twist strands together tightly and curve the wire in the direction that the screw tightens. Saturate the wire with solder to avoid loose strands and ensure tight connections.

Wire nuts can be used for temporary and some permanent connections. The nut must be sized correctly to match the wire size and number. Strip and twist the wires together firmly before applying the nut. Screw the nut on firmly and ensure that it reaches right up to the insulation and leaves no bare wire exposed.



Use of Wire Nut















Soldering – Ensure that connections are clean and well-twisted together before soldering. Always use a flux-core solder. Heat the soldering iron, then clean and “tin” it with a thin film of new solder. Heat the connection to be soldered (do not heat the solder directly) and then apply the solder to the heated connection. This will ensure that the solder will flow well and fill all gaps. Allow the connection to cool before moving it.

























Receptacle Data

Some Common NEMA Receptacle Configurations

Standard Type

 5-15 125VAC	 5-20 125VAC	 6-15 250VAC	 6-20 250VAC	 6-30 250VAC	 7-15 277VAC
 10-20 125-250VAC	 10-30 125-250VAC	 10-50 125-250VAC	 14-30 125-250VAC	 14-50 125-250VAC	 18-60 120-208VAC

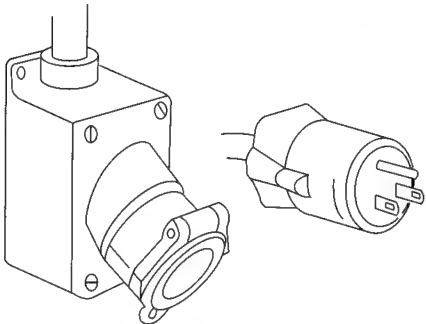
Twist-Lock Type

 ML2-15 125VAC	 L5-15 125VAC	 L5-20 125VAC	 L5-30 125VAC	 L6-15 250VAC	 L6-20 250VAC
 L6-30 250VAC	 L7-20 277VAC	 L8-20 480VAC	 L8-30 480VAC	 ML3-15 125/250VAC	 L10-20 125/250VAC
 L10-30 125/250VAC	 L14-20 125/250VAC	 L14-30 125/250VAC	 L15-20 250VAC	 L15-30 250VAC	 L16-20 480VAC
 L16-30 480VAC	 L18-20 120/208VAC	 L18-30 120/208VAC	 L21-20 120/208VAC	 L21-30 120/208VAC	 L22-20 277/480VAC

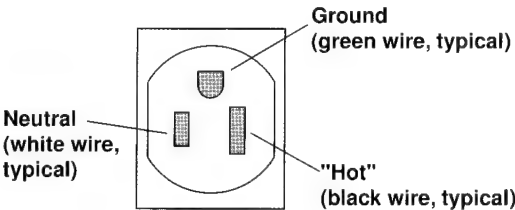
Adapted from [172]

The diagrams below include a UL approved explosion-proof or **hazardous-location receptacle** for single phase equipment. Available for most classes and divisions, these are designed to permit safe connection of equipment in dangerous atmospheres by requiring a “plug and turn” action to activate receptacle power. Also shown is a polarized 3-prong outlet indicating the correct wiring configuration. This convention must never be ignored or serious injury could result. Incorrect wiring could result in electrifying the outside case of the equipment, creating an extremely dangerous situation. Use an outlet tester or a voltage sensor to test for this. Sources [172, 153].

Typical Hazardous Location Receptacle and Wire-Cap



3-Prong Polarized Receptacle



Electrical Enclosure Data

Standard NEMA Electrical Enclosures

NEMA 1 General Purpose	Sheet metal enclosures to protect against dirt and light indirect spraying. Primarily for indoor use.
NEMA 2 Drip Tight	Indoor use to protect against severe condensation and limited amounts of falling water, as in cooling rooms and tunnels.
NEMA 3 Weather Resistant (waterproof)	Outdoor use to protect against splashing rain, sleet, windblown dust, snow or external ice formation.
NEMA 4 Weather Tight	Indoor or outdoor use to protect against windblown dust, direct spraying (must pass 1-inch hose test), and external ice formation. Type 4X includes corrosion protection.
NEMA 5 Dust Tight	Gasketed to protect against falling non-hazardous dust and dirt (not grain, coal or chemical dust) and dripping non-corrosive liquids.
NEMA 6 Submersible	For direct spraying and occasional complete submersion under water for specified time and pressure. Used in mines and quarries.
NEMA 7 Haz. Locations (Class I, Div 1, Groups A, B, C, D)	Indoor use in atmospheres of ether, ethylene, gasoline, benzene, propane, acetone, natural gas and other highly flammable vapors.
NEMA 8 Haz. Locations (Class I, Div 2, Groups A, B, C, D)	Indoor or outdoor use. Used for oil-immersed circuit breakers.
NEMA 9 Haz. Locations (Class II, Div 1, Groups E, F & G)	For combustible dusts of metals, coal, coke, grain.
NEMA 10 Explosion Proof (Bureau of Mines)	For use in coal mines with gassy atmospheres.
NEMA 11 Acid & Fume Resistant, Oil-Immersed, Indoors	For oil-immersed equipment subjected to acid or corrosive fumes. Used in chemical plants, plating operations, etc.
NEMA 12 Industrial	Protects against circulating dust, dirt, oil seepage and dripping of other non-corrosive liquids.
NEMA 13 Dust Proof	Indoor use primarily to protect against dust, water, oil, and non-corrosive coolant. Specially designed for each application.

Adapted from [49, 65, 191, 239]

In addition to the NEMA codes listed above, which are common in the U.S., an alternate system of 2-digit "Ingress Protection" (**IP**) codes, established by the IEC, is widely used outside of North America. The tables below list the definitions of the IP codes and show some example comparisons between NEMA types and their equivalent IP types (note that this table cannot be used to convert from IP to NEMA types). Under the IP system, an enclosure with a designation of IP64 would be dust tight and protected against splashing water. Sources [65, 239].

IEC Ingress Protection (IP) Codes for Electrical Enclosures

First Digit (protection against solid objects)		Second Digit (protection against moisture)	
0	No protection	0	No protection
1	Protected against objects greater than 50mm	1	Protected against dripping water
2	Protected against objects greater than 12mm	2	Protected against dripping water when tilted up to 75°
3	Protected against objects greater than 2.5mm	3	Protected against spraying water
4	Protected against objects greater than 1.0mm	4	Protected against splashing water
5	Dust protected	5	Protected against water jets
6	Dust tight	6	Protected against heavy seas
-		7	Protection against the effects of immersion
-		8	Protection against submersion

Comparison of Some NEMA and Equivalent IP Enclosures

NEMA type	1	2	3	4	5	6	12	13
IP Code	IP10	IP11	IP54	IP56	IP52	IP67	IP52	IP54

Hazardous Location Classifications for Electrical Equipment

Rigorous standards exist for electrical equipment operated in hazardous areas. The National Electrical Code, Article 500 (NEC 500) and Underwriter's Laboratories, Inc. (UL) classify hazardous areas according to the possible presence of an explosive atmosphere due to flammable gases, vapors, or dusts, the likelihood that the explosive atmosphere is present when equipment is operating, and the ignition-related properties of the explosive atmosphere, such as minimum ignition energy and safe electrical air gap dimension. The table below summarizes the system. Sources [65, 238, 239, 251].

Electrical equipment intended for use in hazardous areas, such as explosion-proof motors, lighting fixtures, and receptacles, must be labeled to indicate its suitability for particular types of locations. By far the most common designation encountered in the CPI is for "Class I, Div. 1, Groups C & D" which covers areas in which the most common flammable solvent vapors are expected to be present during normal operations. Equipment officially termed "explosion-proof" and therefore bearing the U.S.-required "Explosionproof" or "AEx" code letters, must meet these criteria (see page 5-12).

The NEC/UL Hazardous Location Classification System

CLASS 1 - FLAMMABLE GASES OR VAPORS			
Division 1 (normal) - Ignitable concentrations exist some or all of the time under normal operating conditions. Acceptable equipment types include explosion-proof, intrinsically safe (2-fault type) and purged/pressurized (types X or Y)			
Division 2 (abnormal) - Ignitable concentrations not likely to exist under normal operating conditions. Acceptable equipment types include: hermetically sealed, non-sparking, oil-immersed, sealed device, purged/pressurized (type Z) and any Class I Division 1 type.			
Group A	Group B	Group C	Group D
Atmospheres containing acetylene.	Atmospheres containing hydrogen (H ₂), fuel or combustible process gases containing more than 30% hydrogen, or of equivalent hazard such as butadiene, ethylene oxide, propylene oxide, and acrolein.	Atmospheres containing highly flammable vapors such as ethyl ether, ethylene, acetaldehyde, allyl alcohol, N-butyraldehyde, CO, crontonaldehyde, cyclopropane, diethylamine, epichlorohydrin, ethylene, ethylenimine, H ₂ S, morpholine, nitropropane, tetrahydrofuran, isoprene, or similar compounds.	Atmospheres containing flammable vapors such as acetone, ammonia, benzene, butane, butanol, butyl acetate, ethane, ethanol, ethyl acetate, gasoline, heptane, hexane, methane, methanol, MEK, MIBK, natural gas, pentanes, propane, propanol, propylene, toluene, xylene or compounds of similar flammability.

CLASS II - COMBUSTIBLE DUSTS		
Division 1 (normal) - Ignitable concentrations exist some or all of the time under normal operating conditions. Acceptable equipment types include dust-ignition-proof, intrinsically safe and pressurized.		
Division 2 (abnormal) - Ignitable concentrations not likely to exist under normal operating conditions. Acceptable equipment types include: dust-tight, non-sparking, pressurized and any Class II Division 1 type.		
Group E	Group F	Group G
Atmospheres containing combustible metal dusts, such as aluminum, magnesium and their alloys, or other combustible dusts whose particle size and conductivity present similar hazards.	Atmospheres containing combustible dusts of coal, coke, carbon black and charcoal, dusts containing more than 8% volatiles or dusts that present a similar hazard.	Atmospheres containing other combustible dusts such as flour, starch, grain, cocoa, wood, plastic and chemicals.

CLASS III - EASILY IGNITABLE FIBERS OR FLYINGS
Division 1 - Where easily ignitable fibers or materials producing combustible flyings are handled, manufactured or used. Acceptable equipment types include intrinsically safe and dust-tight.
Division 2 - Where easily ignitable fibers are stored or handled. Acceptable equipment types include intrinsically safe and dust-tight.
No separate groups specified. Materials include rayon, nylon, cotton, sawdust, woodchips and similar fibrous substances which are easily ignitable, but not apt to be present in the air in sufficient quantities to produce ignitable mixtures.

NEC Equipment Maximum Surface Temperature Codes (°C)

T1	T2	T2A	T2B	T2C	T2D	T3	T3A	T3B	T3C	T4	T4A	T5	T6
<450	<300	<280	<260	<230	<215	<200	<180	<165	<160	<135	<120	<100	<85

An additional marking found on hazardous-area equipment is a **temperature code letter**, which indicates the maximum external surface temperature that the equipment is expected to reach during normal operations. The table above lists the temperature codes used by the NEC.

IEC and NEC 505 Zone and Classification System – For countries outside North America, standards and classifications for hazardous area equipment are set by the IEC/CENELEC, based on a different system. And although the NEC 500 is still the most widely used in the US, an IEC-like system is also being introduced beginning with the adoption of NEC Article 505 in 1996. Instead of Division classes, the IEC and NEC 505 use *Zones* and *Explosion Groups* to indicate the likelihood that an explosive atmosphere will be present while the equipment is in operation. Under this system, three zones are identified: **Zone 0** – where ignitable concentrations of flammable gases, vapors or liquids are *present continuously* or for long periods of time under normal operating conditions, **Zone 1** – where ignitable concentrations of flammable gases, vapors or liquids are *likely* to exist under normal operating conditions, and **Zone 2** – where ignitable concentrations of flammable gases, vapors or liquids are *not* likely to exist under normal operating conditions.

Confusion can easily arise from the additional group classifications used by the IEC which are ordered in reverse of the group designations employed in the US. The table below is included to allow a comparison of some typical equipment markings in both the NEC/UL and IEC systems. The IEC likewise uses maximum surface temperature codes, but does not subdivide the temperature ranges by using letter suffixes in the codes. Their designations are limited to the six codes T1 through T6.

Some Representative NEC/UL and IEC Hazardous Area Equipment Designations

	NEC/UL Marking	Alternate or Additional IEC Marking
Intrinsically Safe Equip.	UL Class I, Division 1, Group D	Class I, Zone 0, Group IIA
	UL Class I, Division 1, Group C	Class I, Zone 0, Group IIB
	UL Class I, Division 1, Group B	Class I, Zone 0, Group IIB plus H ₂
	UL Class I, Division 1, Groups A & B	Class I, Zone 0, Group IIC
Any Other Equipment	UL Class I, Division 1, Group D	Class I, Zone 1, Group IIA
	UL Class I, Division 1, Group C	Class I, Zone 1, Group IIB
	UL Class I, Division 1, Group B	Class I, Zone 1, Group IIB plus H ₂
	UL Class I, Division 1, Groups A & B	Class I, Zone 1, Group IIC
	UL Class I, Division 2, Group D	Class I, Zone 2, Group IIA
	UL Class I, Division 2, Group C	Class I, Zone 2, Group IIB
	UL Class I, Division 2, Group B	Class I, Zone 2, Group IIB plus H ₂
	UL Class I, Division 2, Groups A & B	Class I, Zone 2, Group IIC

Sources: [65, 238, 239, 251]

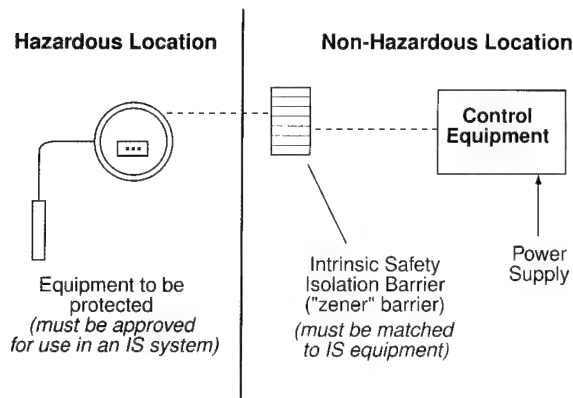
Examples of Typical Explosion-Proof Equipment Markings

NEC 500	Explosionproof with I.S. Outputs, Class I, Division 1, Groups A, D, C, D, T5	
NEC 505	Class I, Zone 1, AEx d [ia] IIC T5	d = protection method (d - flame-proof enclosure, e - increased safety, p - pressurized apparatus, i - intrinsically safe, o - oil-immersed)
CENELEC	Ex d [ia] IIC T5	[ia] = IS output (ia - double redundancy, ib - single redundancy)
IEC	EEx d [ia] IIC T5	II = Group C = Gas group T5 = Temperature class

Intrinsically Safe Equipment

Intrinsic safety (IS) is an explosion prevention method used for equipment and wiring in hazardous locations that can provide an economical alternative to explosion-proof equipment and purged enclosures in certain situations. IS equipment is designed so as to be incapable of releasing sufficient electrical or thermal energy to ignite a surrounding explosive atmosphere under any circumstances. This is accomplished by using devices (called intrinsic safety isolation barriers) that limit the amount of power available to equipment in the hazardous location below the level of possible ignition, thereby rendering it inherently or *intrinsically* safe for use. The U.S. standards for such systems are established by ANSI and UL, based on careful assessment of the minimum required ignition energy of any given vapor/air mixture.

Typical Application of an Intrinsically Safe System



It is understood that when properly installed, the IS system will prevent ignition regardless of the condition of the circuit. That is to say that it is designed for the worst possible case, wherein the circuit has failed, shorted, grounded or is exposed to a higher voltage than that for which it was designed.

As a general rule, the use of intrinsic safety is limited to systems that use less than 1 watt of power. Higher energy circuits require other means of explosion protection. Nor is it designed to protect against electrostatic sparks or lightning strikes. Nonetheless, intrinsically safe designs are approved for many devices such as thermocouples, pressure and level switches, solenoid valves, and 4-20mA transmitters.

In a typical application, the hazardous location field device or equipment is isolated from the power supply by means of an

intrinsic safety isolation barrier (often called a “zener” barrier) that is placed in a safe or non-hazardous area. The barrier limits the amount of electrical energy that can enter the hazardous area to intrinsically safe levels. The field equipment must be approved for use in the intrinsically safe system and the barrier must be approved for and matched to the equipment in use. Another common type of isolating device is the **galvanic isolator**.

Field apparatus can be divided into two types. **Simple** devices are those that operate at such low energy levels that they cannot ignite a flammable atmosphere under fault conditions, and will not store more than about 1.2 V or 0.25 mW of energy. These include thermocouples, RTD's, LED's and some switches. These devices can be connected to a certified IS circuit without the evaluation of an approved testing agency. Devices that operate at energy levels higher than simple devices such as transmitters, solenoid valves and electric actuators are considered to be **energy storing** devices, and do require testing agency approval for use in an intrinsically safe system.

Energy storing devices are evaluated for use in IS circuits based on a number of key criteria such as the maximum possible open-circuit voltage, the maximum short-circuit current, minimum resistance, the maximum capacitance that may be discharged under fault conditions, and the maximum voltage and current the device can withstand before component failure results in dangerous levels of heat.

Equipment and protective devices are often approved together as a set, with precise identification including manufacturer and model number and specific information about the proper connection and use of the devices. This is called a “loop approval”. In other cases, devices are assigned parameters that allow them to be properly matched with a protective device to produce a complete intrinsically safe system. This is called an “entity approval”.

Wiring characteristics must also be considered when designing intrinsically safe installations. Wire itself is capable of generating induction currents or releasing stored capacitance as a spark, and so igniting a flammable vapor. It is recommended that wire with a capacitance (per wire pair) of no more than 60 pF/ft and maximum inductance of 0.2μH/ft be used in IS systems [239]. It is also critical that all IS circuits be isolated from any non-IS circuits.

There are many reputable manufacturers of IS equipment and devices, including Omega Engineering and Stahl. It is wise to discuss with them the specifics of your application and any particular concerns you may have. Sources [65, 191, 239].

Electric Motors

Electric motors convert electrical power to rotary motion through the action of magnetic fields, one on the rotating member (rotor) and one on the stationary member (stator), which are in a particular spatial position relative to one another. Motors fall into two major types, direct current (DC) and alternating current (AC). In **DC** motors, the rotor magnetic field is generated by direct current passed through a winding on the rotor (called a *field* winding), transmitted by brushes or slip rings. If powered from the AC electrical grid, a power converter is required. DC motors are also relatively high maintenance and are not widely used in the CPI.

AC motors can also be divided into two types, synchronous and induction (asynchronous). In **synchronous** motors, the rotor magnetic field is produced by permanent magnets on the rotor or by current passing through a field winding via brushes. Synchronous motors are always polyphase, and always run at a single fixed steady-state speed determined by the stator frequency. In **induction** motors, which may be either single phase or polyphase, the rotor magnetic field is *induced* by current passing through windings on the stator. Induction motors are by far the most common.

Smaller induction motors, up to 5 or 10 HP, usually operate on **single-phase** power while larger motors are usually **three-phase**, but there is considerable overlap. In three-phase motors, a rotating magnetic field exists at power-up (called the locked-rotor state). Single-phase motors have no rotating field at start-up and thus generate no start-up torque without the use of an auxiliary winding and capacitor to get the motor turning and a switch to disconnect the auxiliary after starting. This, and lower power transmission losses, make three-phase motors more efficient and reliable than single-phase and they are much more widely used. Three-phase motors are connected to three separate voltage legs, with appropriate overcurrent protection, such as a circuit breaker, on each one. The direction of a three-phase motor can be reversed by switching any two of the three wire leads to the motor.

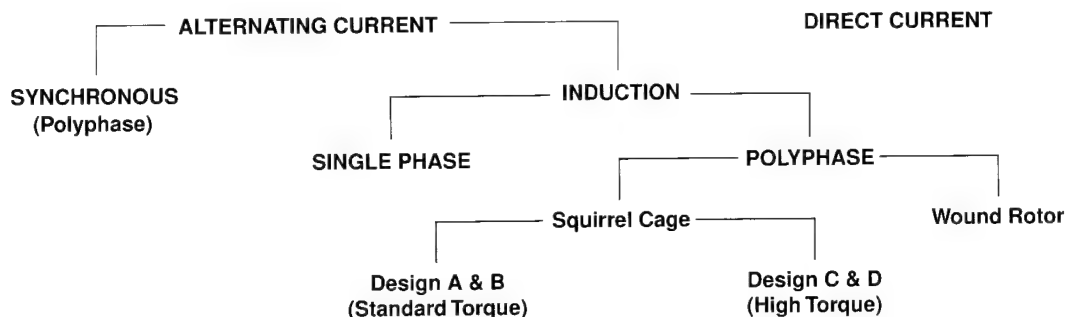
Motor Nameplate Data

Along with the name of the manufacturer, model and serial number, a variety of important information is listed on motor nameplates. A number of the key terms and their significance are listed below.

Horsepower rating indicates the motor's maximum normal power output. NEMA has established a number of standard horsepower-rated motors. Outside of North America, it is common to see motors rated in kilowatts (1 HP = 0.75kW). The typical current (amps) drawn by various sized motors is listed in the table on page 5-12.

Voltage – Operating voltage is a key motor design parameter. Common nominal voltages for three-phase motors are 208, 240, 480 and 600V (and the rated voltages corresponding to these are 200, 230, 460 and 575V). When voltage is too high or too low the motor may perform poorly and have a reduced life. In reality, few motors operate at exactly their rated voltage. NEMA specifies that a motor should be designed to run within $\pm 10\%$ of its rated voltage, but for the best efficiency and performance, the voltage should be kept within $\pm 3\%$. Some motors are designed to operate at several voltages. For example, the 208/230-460 “tri-voltage” type may be wired for either 230 or 460V, and when wired for 230 it can accommodate 208. But, here again, such a motor operating at 208V will run hotter, have lower efficiency, produce less torque and have a shorter life than a motor designed for 200V.

Classification of Electric Motors



Typical Full-Load Current Draw of Induction Motors (in amperes)

Single Phase				Three-Phase				
HP	115V	208V	230V	HP	115V	208V	230V	460V
1/6	4.4	2.4	2.2	1/2	4	2.2	2	1
1/4	5.8	3.2	2.9	3/4	5.6	3.08	2.8	1.4
1/3	7.2	4.0	3.6	1	7.2	3.96	3.6	1.8
1/2	9.8	5.4	4.9	1-1/2	10.4	5.72	5.2	2.6
3/4	13.8	7.6	6.9	2	13.6	7.48	6.8	3.4
1	16	8.8	8	3	-	10.56	9.6	4.8
1-1/2	20	11.0	10	5	-	16.72	15.2	7.6
2	24	13.2	12	7-1/2	-	24.2	22	11
3	34	18.7	17	10	-	30.8	28	14
5	56	30.8	28	15	-	46.2	42	21
7-1/2	80	44.0	40	20	-	59.4	54	27
10	100	55.0	50	25	-	74.8	68	34
				30	-	88	80	40
				40	-	114.4	104	52
				50	-	143	130	65

Sources: [87, 107, 161, 167]

Frequency – Motors are also designed to operate at a specific frequency. AC frequency in the US is 60 Hz but in many places it is 50 Hz. Operating a motor more than $\pm 5\%$ outside of its rated frequency can seriously affect its performance.

Efficiency and Power Factor – Motor efficiency is the percentage of the electrical energy that winds up performing useful work. A difference in efficiency of a few percentage points can make a significant difference in the operating cost of a motor, which can, over the 10-15 year lifetime of the motor, consume an amount of electricity equal to more than 100 times the initial motor price. Peak efficiency for most motors actually occurs at about 3/4 of the rated load. Operating a motor below 50% of its rated load can result in extremely low efficiency. Thus it is important to not *oversize* a motor for a given duty. The power factor is a bit more obscure. It is a measure of how much of the power drawn is used to induce the magnetic field that drives the motor, and indirectly reflects the overall motor system efficiency.

Service Factor – This is an indication of how much above its rated horsepower a motor can operate for short periods without failing. Higher power factors usually mean that the motor's insulation materials can withstand somewhat higher temperatures than specified. Power factors can range up to 1.15 for new motors, but operating them continually above their rated loads results in reduced efficiency and shortens the motor's life. A high service factor should be considered more as an indication of a high quality motor and not a license to operate a motor at overcapacity. A motor must be *derated*, or given a service factor below 1.0, if it is to operate in harsh or high-temperature environments.

Motor Code – This is a letter indicating the maximum current the motor will draw (in kilovolt amperes per rated horsepower) at start-up. This is often called the "locked-rotor kVA value". These codes are used when sizing power lines, circuit breakers, etc. Some common codes and their values are listed in the table below.

Enclosure – NEMA motor enclosure standards are based on environmental conditions. The two general classifications are **open** and **totally enclosed**. An open motor has ventilation openings to permit external air to cool motor windings. A totally enclosed motor is constructed to prevent the exchange of air between the inside and outside, but not sufficiently so as to be airtight. Some of the common types of motor enclosures are listed in the table on page 5-13. In many cases there is no safe option but to use totally enclosed motors to prevent fires in hazardous environments. **Explosion-proof** motors are a special class of totally enclosed motors designed to withstand explosions *within*, to prevent sparks from igniting a flammable atmosphere, and to maintain an external temperature that will not ignite the surrounding vapors. Typical designations are "approved for Class I, Group D", or "Class II, Groups F & G", which refer to the NEC classification system for hazardous environments, described in more detail on page 5-8.

Some Common Locked-Rotor kVA Codes

(kilovolt-amperes per rated horsepower, kVA/HP)

A	0-3.15	E	4.5-5.0	J	7.1-8.0
B	3.15-3.55	F	5.0-5.6	K	8.0-9.0
C	3.55-4.0	G	5.6-6.3	L	9.0-10.0
D	4.0-4.5	H	6.3-7.1	M	10.0-11.2

To estimate actual motor starting current:

$$\text{Amps} = \frac{1000 \times \text{rated HP} \times \text{kVA/HP}}{1.73 \times \text{volts}}$$

Common Types of Electric Motor Enclosures (NEMA)

Open	No air-flow restriction, used where motor will not be exposed to water, dust or chemicals, and out of the way of personnel. Usually with internal cooling fan.
Drip-Proof (DP)	Protected from liquids or solids falling from a vertical position.
Encapsulated	Drip-proof type with the stator windings completely surrounded by a protective coating, offering more resistance to moisture and corrosive environments.
Guarded	All openings small enough to prevent insertion of fingers, etc. to live or moving components
Totally Enclosed Fan-Cooled (TEFC)	Sealed to prevent exchange of air from within and outside the motor. Usually have an external shaft-mounted cooling fan. For use in wet or dusty locations.
Totally Enclosed Non-Ventilated (TENV)	Sealed as a TEFC, but for environments too dirty to allow the use of an external cooling fan. They thus do not dissipate heat as well as TEFC.
Explosion Proof (XP)	Totally enclosed to prevent sparks or flames within the motor from igniting flammable vapors outside, and also designed to withstand an explosion of a gas or vapor inside the motor.

Sources: [87, 161, 251]

Temperature and Insulation Class – Most industrial motors are rated to operate in a 40°C environment. Their service factors should be reduced if this condition is exceeded. Increased operating and ambient temperatures can damage bearings and shorten the life of the insulation on the windings, resulting in premature failure of the motor. There are four NEMA insulation classes – A, B, F, and H – each with increasing temperature rating. They consist of a maximum allowable ambient temperature and temperature rise with allowances for hot spots and service factors. Class B insulation is standard and allows for a total temperature of 130°C (40°C ambient plus a rise of 70°C for ODP motors and 75°C for TEFC motors). Class F (maximum temperature rating of 145°C) or higher is recommended for new motors.

Frequent starts, overloading, improper power source, high altitude, poor ventilation, dirt or sunlight can also cause overheating. Many motors have **thermal protection** devices that cut power to the motor if a maximum temperature is exceeded. Over-temp protection ‘1’ prevents the windings from exceeding the insulation’s temperature rating at start-up; ‘2’ offers protection during continuous full-load conditions. These devices are available with manual or automatic reset. Automatic reset must be used only where automatic motor restart will not impact process or personnel safety.

NEMA also sets standards for the various types and sizes of motor **frames** or mountings. Motors may be foot-mounted, face-mounted or flange-mounted. Horizontal foot mounting is the most common, but motors can also be foot-mounted on a vertical surface with the shaft up, down or running horizontally. The table and figure on page 5-14 list some of the more common motor foot mounting dimensions. Frame codes are often followed by additional characters, as in 213T or 254U. These letters refer to various motor shaft configurations (diameter, length, keyslot dimensions). International motor frame standards are specified by the IEC (International Electrotechnical Commission).

Design Type refers to which of the basic NEMA design groups the motor complies (A, B, C, D, wound rotor or multi-speed) each of which is characterized by varying torque, slip, power ratings and other factors. By far the most common is the three-phase squirrel-cage style Design B induction motor with moderate torque, starting current, and 3% slip.

Motor Speed is determined by the frequency of the AC power (60 Hz in the US and 50Hz in many other countries) and the number of stator windings. Nominal speeds of most motors are either 900, 1200, 1800, or 3600. These values represent the *synchronous* speed, or the rotational speed of the magnetic field. The actual motor speed is slightly lower than this due to the phenomenon known as *slip*. Thus a typical Type B three-phase motor with a synchronous speed of 1800 and a slip of 3% actually operates at 1745 rpm. Changes in temperature and line voltage can also affect motor speed, thus if precise speed control is necessary, it is best accomplished with a **variable frequency drive (VFD)**.

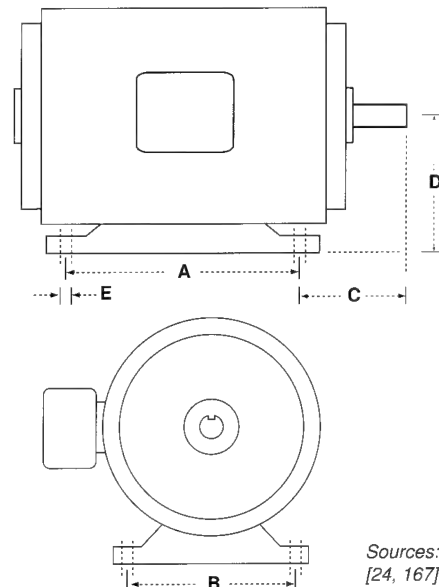
Motor Control devices must be selected carefully to fit the overall operating and control strategy of the plant, and they must be properly installed and operated if their full potential and benefits are to be realized. Some of the important types of motor controllers are listed here: **Across the line starters** – manual on/off switches that connect the motor directly to the supply at the rated voltage without using resistance or autotransformers to reduce the starting voltage. These may include thermal and surge protection. **Magnetic starters** are also usually manually controlled, but they allow the operation of a motor that is not in the immediate vicinity. The switch energizes magnetic contacts at a remote location that start the current to the motor. They often include overprotection circuitry. **Duty cycle controllers** – typically consist of a system of switches that automatically control motor on/off based on time, remote sensors or other parameters. **Soft-start**

controllers – control devices that reduce the high surge of current at start-up by adjusting voltage, impedance, use of partial windings or other means. Start-up current can reach several times the normal operating current and trip circuit breakers, cause power dips and motor stress. **Torque controls** – power saving devices that reduce torque at times of low load while maintaining motor speed. **Speed controls** – these systems can greatly cut energy costs and improve equipment life. Options include multi-speed motors (speed depends on wiring configuration), mechanical gear drives, and electronic drives such as variable frequency drives that reform the 60 Hz power cycle into different frequencies. Since they consume power, such units are often bypassed when operating the motor at full speed.

Electric Motor Troubleshooting Guide									
Possible Cause ↓	Symptom →	Motor won't start	Motor stalls	Frequent blown fuses or breaker	Motor speed too low	Motor accelerates slowly	Motor draws excessive current	Motor turns in wrong direction	Motor overheats
Blown fuse or tripped breaker		x							
Open circuit in wiring		x	x		x				x
Incorrect power supplied		x							
Motor connections incorrect		x				x			
Starter circuit problem		x							
Inappropriate motor selection			x		x				
Motor mechanically frozen		x							
Motor overloaded		x	x		x	x			x
Stator winding defective/shorted		x		x			x		x
Rotor defective		x			x	x			
Voltage too low			x		x	x			
Voltage too high							x		
Wrong phase sequence (switch 2 of 3 leads)								x	
Insufficient motor cooling									x
Unbalanced voltage									x
Failing bearings, shaft misaligned									x
Rotor or fan rubbing									x
Excessive belt tension									x
Unbalanced rotor									x
Pulley undersized									x
Improper/insufficient lubrication									x

Some NEMA Standard Motor Frame Sizes
(all dimensions in inches)

NEMA#	A	B	C	D	E
48	2.75	4.25	4.00	3.00	
56	3.00	4.88	4.63	3.50	
66	5.00	5.88	5.38	4.13	
143	4.00	5.50	2.25	3.50	0.34
145	5.00	5.50	2.25	3.50	0.34
182	4.50	7.50	2.75	4.50	0.41
184	5.50	7.50	2.75	4.50	0.41
213	5.50	8.50	3.50	5.25	0.41
215	7.00	8.50	3.50	5.25	0.41
254	8.25	10.00	4.25	6.25	0.53
256	10.00	10.00	4.25	6.25	0.53
284	9.50	11.00	4.75	7.00	0.53
286	11.00	11.00	4.75	7.00	0.53
324	10.50	12.50	5.25	8.00	0.66
326	12.00	12.50	5.25	8.00	0.66
364	11.25	14.00	5.88	9.00	0.66
365	12.25	14.00	5.88	9.00	0.66
404	12.25	16.00	6.63	10.00	0.81
405	13.75	16.00	6.63	10.00	0.81
444	14.50	18.00	7.50	11.00	0.81
445	16.50	18.00	7.50	11.00	0.81
504	16.00	20.00	8.50	12.50	



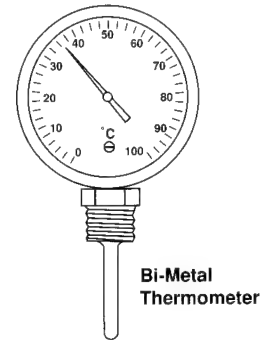
Sources:
[24, 167]

Temperature Measurement

A tremendous variety of temperature measurement instruments are in use, including non-contact infrared and IC sensors, liquid crystals and ultrasonic sensors. Here we will focus on those types most commonly used in the CPI.

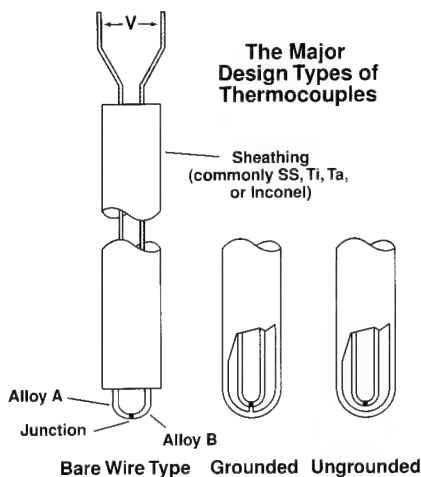
The familiar liquid-filled glass thermometer receives only brief mention here, because although still widely used in laboratory situations, there is little call for its use in pilot plants or manufacturing facilities. Indeed, with the wide availability of fast, accurate digital thermometers, there is really little reason to continue its use in most situations. This is particularly true of mercury thermometers whose contents represent a serious environmental hazard.

Bimetal thermometers are extremely dependable, passive devices that can be safely used for local temperature readings in virtually any environment. It is often wise to back up the more sophisticated electronic sensing systems with bimetals for local temperature indication because of their simplicity. The heart of these instruments is a coil or spring consisting of two strips of different metals bonded together. The two metals have different expansion coefficients. As the temperature increases, the coil bends, deflecting a needle or other indicating device. They are reasonably accurate, usually within 1% of full scale, as long as they are periodically calibrated, and they have a rather wide temperature sensing range of about -100°C to 500°C . They must be selected carefully based on material compatibility, pressure rating, location, and other factors. They are often inserted into thermowells for higher pressure or corrosive service and for ease of replacement and calibration. Such thermowells may need to be filled with silicone oil or similar fluid to improve response time.



The remaining devices used in the CPI are principally thermocouples and resistance temperature detectors of which there are two types: simple RTDs and thermistors. These are electronic devices that are wired to an appropriate meter or transmitter to enable remote temperature measurement and control. They can be used in hazardous locations, as long as they are paired with a proper intrinsically safe isolation barrier and meter.

Thermocouples (TCs) are probably the most widely used electronic temperature measurement devices because of their versatility, durability and low cost. They are passive, zero-current devices, requiring no outside source of power. The sensing element consists of the junction of two different metallic alloys. As the temperature of this junction changes, a small thermoelectric voltage is induced between the other ends of the leads attached to the junction (this is called the Seebeck effect). The voltage change is not linear and it varies depending on the junction materials, but that is compensated for by the digital thermometer to which the TC is attached. The digital thermometer is essentially a voltmeter that interprets the voltage change, compares it to an internal electronic reference, and displays it as a temperature reading. This makes the use of TCs simple, but it is critical that the digital meter be correctly matched to the particular type of thermocouple.



There are many thermocouple types, classified by design style and junction material type. The three principle design styles (bare wire, grounded and ungrounded) are shown in the diagram to the left. Bare wire types are almost never seen in the CPI, since the junction needs to be protected from numerous environmental elements by sheathing in a metallic, ceramic or PTFE sleeve. The grounded and ungrounded types generally give more stable readings anyway, since the metallic sheath isolates the device from electronic noise. The grounded type, in which the junction contacts the sheath, is slightly faster responding, but the response time is a matter of only seconds in any case. The choice really depends on the instrumentation.

The other primary distinction between TCs is material type. The alloy combinations used are carefully matched, as are the special thermocouple alloy wires that must be used to connect to the meter or controller. The most common TCs are types J, K, T and E. The characteristics of these types are given in the table on page 5-16. Note that Constantan is not an alloy but rather a family of Cu-Ni alloys. TCs are capable of measuring very broad temperature ranges. The

Common Thermocouple Types

Type	J	K	T	E
Alloys	[Iron] : [Constantan]	[Ni/Cr] : [Ni/Al-Si]	[Cu] : [Constantan]	[Ni/Cr] : [Constantan]
T Range °C	0 to 760	0 to 1370	-160 to 400	-100 to 1000
Accuracy	± 0.1°C	±0.7°C	±0.5°C	±0.5°C
Response Coeff.	50 µvolts/°C	40 µvolts/°C	38 µvolts/°C	60 µvolts/°C
Color Codes*	Black	Yellow	Blue	Purple

*negative lead is always red

Source: [191]

selection of a particular type, along with a specifically type-matched meter or transmitter and intrinsic isolation barriers if necessary, should be made with the help of a qualified vendor such as Omega Engineering, based on temperature range, chemical resistance, the type of measurement you are making and the environment and location of the instrument.

RTDs – resistance temperature detectors are based on the principle that the electrical resistance of metals and other materials is a function of temperature. By passing a small current through a thin wire of a given material and measuring the resistance, the temperature can be determined. Thus, RTDs are active, not passive devices and they require an outside power source to generate the measuring current. They are more sensitive, more stable and somewhat more costly than thermocouples.

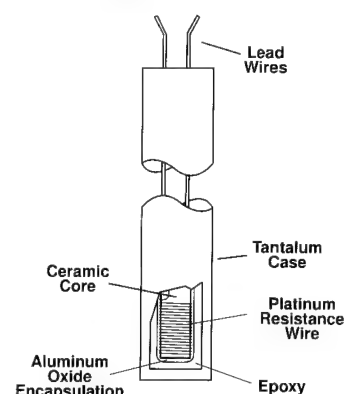
The sensing element of most RTDs consists of a thin wire of known resistance wrapped around a non-conducting core and coated with glass or ceramic, although other construction techniques exist. Resistances range from roughly 10 to 1000 Ω (ohms). By far the most common is the 100 ohm platinum RTD (PRTD 100), although Ni or Ni alloys are also used. It offers accurate, stable measurements with little long term drift (decalibration). The change in resistance is quite linear, with an average slope of 0.00385 Ω / °C between 0 and 100°C. Since the intrinsic resistance is rather low, the resistance imparted by the long leads connected to the meter or control apparatus can introduce measurement error as can the actual current flowing through the sensor.

However, these errors are greatly minimized by the use of correctly matched RTD wire and a properly installed sensor. 2-, 3-, and 4-wire RTDs are found, with the 3rd and 4th wires used to provide precise compensation for greater accuracy. With well-matched wires, however, the 2-wire RTD provides sufficient accuracy for most needs.

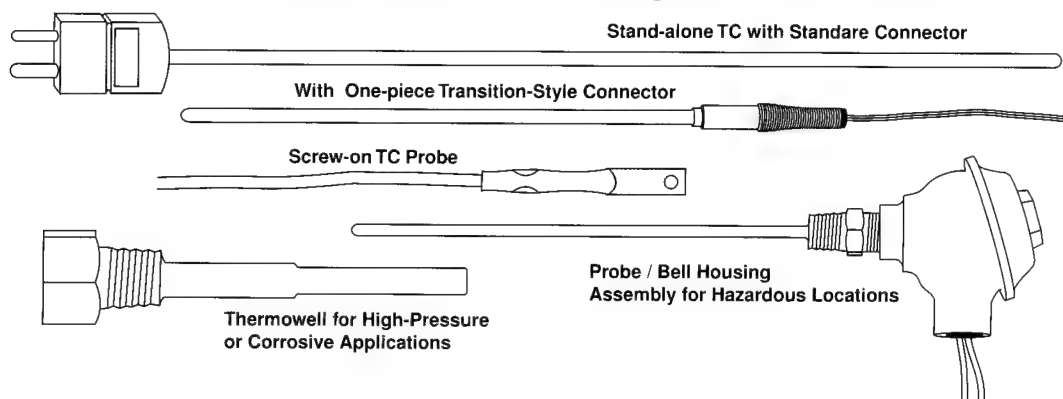
The temperature range of RTDs is not quite as broad (roughly -250 to 900°C), but is certainly sufficient for CPI operations. RTDs are also somewhat more fragile than the rugged thermocouples. They should be handled with care.

Thermistors operate in same way as RTDs, but the resistance element is made of a synthetic semiconductor. They have the highest accuracy, but are also the most costly and most fragile. They are designed to have a much higher intrinsic resistance (typically about 5000Ω) and a resistance change of up to 3-4% per °C. This makes any resistance imparted by

A Typical 2-Wire RTD Sensor Tip



Some Typical Temperature Sensor Configurations and Assemblies



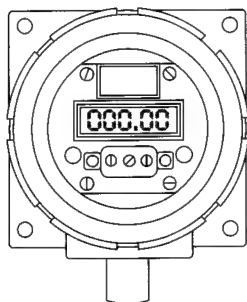
long lead wires insignificant, and renders the device highly sensitive, capable of measuring very minute changes in temperature, although the temperature range is more limited than RTDs (-400 to 150°C). Thermistors are more widely used in laboratory and specialty applications requiring very high accuracy or sensitivity.

Installation and Use – Since the complete electronic temperature measuring system is composed of many components, the detector, the wiring, isolation barriers or field transmitters, signal converter or meter and controls, it goes without saying that the installation should be supervised by someone qualified to do so, particularly in hazardous locations. Likewise, the system must be fully tested and checked for proper calibration prior to use. Most reputable vendors of electronic temperature sensors and controls can provide invaluable advice and assistance in the selection and matching of components as well as installation and testing. The Omega Engineering Temperature Catalogue/Handbook for example, available for free, is a veritable encyclopedia of temperature measurement technology. It is definitely wise to diagram the proposed installation in detail and have someone knowledgeable in the field look at it before ordering parts or beginning installation.

Some Key Things to Remember – Wires used to run from the sensor to the controls must be approved twisted-pair thermocouple or RTD wire types. Always use consistent wiring, shielded and of large diameter to minimize noise and ensure proper operation. Avoid any mechanical stress on the leads. Meters, barriers and other equipment must be matched to the particular type of detector for proper operation. Since most TCs and RTDs are considered simple non-energy-storing devices, they do not need to be approved before use in an intrinsically safe system, but they must be properly matched to isolation barriers (see page 5-10). Finally, once installed, the system must be fully tested and then scheduled for regular periodic checks, calibration and preventive maintenance.

The 4-20 mA Transmitter

Signals produced by field instruments such as thermocouples, RTD's, and pH probes are very low in signal strength. If the leads run any significant distance to controls or isolation barriers, the voltage drop due to resistance and electronic noise or temperature changes along the way can affect the signal and compromise accuracy. The industry-wide standard for overcoming this problem is amplification of the signal by converting it to a current flow, between 4 milliAmps and 20 milliAmps. The current will not change appreciably over long distances. Once at its destination, the current "signal" is converted back to a voltage by means of a resistor for easier interpretation by the meters or controls as needed.



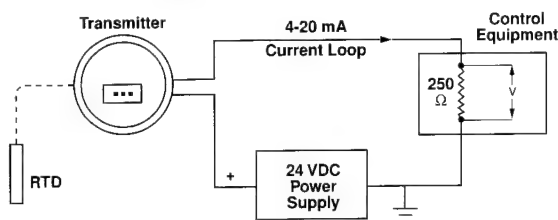
Typical 4-20mA Transmitter

In this system, 4 mA is considered the "live" zero, equivalent to the bottom of the instrument's range and 20 mA is set to the top of the range. A true zero current situation would indicate a system failure. At the control panel, the current is passed through a resistor, usually 250Ω , that converts the current to a voltage that is interpreted by the appropriate circuitry as temperature. By Ohm's law (page 5-4), 4-20mA passing through a 250Ω resistor results in a voltage of 1-5 volts. Thus, for a TC whose range is 0° to 200°C , 4 mA (or 1V) represents 0° and 20 mA (or 5V) represents 200° . The scale is linear.

The figure above shows a typical 4-20 mA transmitter, totally enclosed for use in hazardous locations. This particular type is called an "indicating" transmitter since it provides a local reading as well as converting the instrument signal to the appropriate current and setting up the "current loop" to and from the control system. 4-20 mA transmitters require an external source of power, usually 24 VDC. A very simplified schematic is shown at right. Sources [69, 191].

The selection of a transmitter must be based on the nature of the field instrument, the type of signal being processed, the resistance load of the complete circuit, and the environment in which the transmitter will be located. For hazardous locations, transmitters must be rated as explosion-proof, or must be approved and certified for inclusion in an IS system.

Typical Application of a 4-20 mA Transmitter



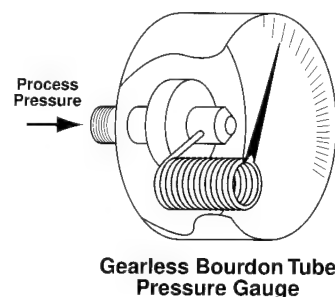
Pressure Measurement

Pressure sensing devices consist of a whole array of instruments ranging from simple mechanical pressure gauges to sophisticated electronic sensors based on changes in capacitance or resistance and capable of communicating pressure data to remote control equipment as 4-20 mA or other signals.

The workhorses for local pressure indication are the **mechanical dial gauges** that convert pressure change into the movement of a pointer via a bellows, diaphragm, Bourdon tube or similar element. The accuracy of such devices varies (see the classification table at the bottom of the page) and their calibration should be checked regularly. Positive pressure, vacuum, and compound gauges (reading pressures both above and below atmospheric pressure) are widely available in many sizes and ranges. Liquid-filled dials (usually glycerin-filled) are often seen. The glycerin in this case does more than dampen pointer vibration. By keeping out ambient air, it eliminates the possibility of moisture condensation inside the case – a very common cause of gauge failure because of the corrosion it can cause and the possibility of freezing in outside installations. Other gauge options include illuminated dials, separate pointers to record the minimum or maximum pressure reached (a useful troubleshooting tool), or the more expensive digital local gauges that provide a numerical readout on a display, many of which are battery operated.

Precautions – Pressure gauges must be selected and installed so as to minimize the possibility of injury or damage from failure, especially those used in hazardous service such as oxygen or other high pressure gas. Consult the manufacturer for specific information about your application. In general, the primary considerations are pressure, vibration, pulsation, temperature range and material compatibility. Select gauges so the range is twice that of the expected operating pressure, and never pressurize a gauge beyond the top of its scale. Vibration and pulsation can cause wear and diminish accuracy. Thus, gauges should be isolated by means of appropriate protective devices (page 5-19). Service standards for gauges appear in ASME bulletin B40.1 [21].

The figure at the right shows a typical gearless Bourdon tube pressure gauge movement. Such devices are simple, economical, and sufficiently accurate for most process purposes. They are very reliable because of their few moving parts.



Reference Pressure – All pressure devices must report data with respect to a reference pressure and it is important to consider this for your application. *Absolute* pressure gauges (which read in **psia**, for example) use absolute vacuum as their reference (although absolute vacuum is not attainable, introducing a minor error for high vacuum applications). *Gauge* pressure instruments (reading in, say, **psig**) have atmospheric pressure as their reference. Atmospheric pressure can vary by as much as 0.5 psi, thus introducing another small error, particularly in compound or low-pressure gauges. Differential pressure gauges have no reference pressure per se since they are connected to two different process pressure points and measure the difference between them. The designation “**psid**” is sometimes used to express these readings.

Principles of Operation – The most common types of sensing methods are briefly described here to familiarize the reader with the basic terminology and operating principles. **Bourdon tubes** (see figure above) are one of the most widespread, versatile and inexpensive sensor types. The heart of the device is a thin, partially flattened metallic tube that is connected directly to process pressure at one end and to a pointing device at the other. Picture a child's roll-up party favor. As pressure increases, the tube, usually bent in the shape of a C or a spiral, tends to straighten, resulting in mechanical movement at the pointer end.

Bellows-based Gauges are based on the expansion or contraction of a small accordion-like bellows usually made of thin

Pressure Gauge Accuracy Classification

ASME accuracy grade	Permissible error (% of full scale)	Minimum recommended face dial diameter	ASME accuracy grade	Permissible error (% of full scale)	Minimum recommended face dial diameter
D	5	1.5"	1A	1	1.5"
C	4	1.5"	2A	0.5	2.5"
B	3	1.5"	3A	0.25	4.5"
A	2	1.5"	4A	0.1	8.5"

Sources: [47, 64]

brass, bronze, stainless steel or specialty alloys. $\pm 2\%$ accuracy is typical, and can be much higher ($\pm 0.2\%$) in double-bellows models that can compensate for changes in atmospheric pressure. These are generally more expensive than Bourdon-tube-types.

Diaphragm Sensors – These measure pressure by the movement of a flat diaphragm exposed to the process pressure. Modern diaphragms are made of many materials, most notably beryllium-copper or Ni-Span C. Stainless steel or Inconel are used for corrosive service with a slight compromise in accuracy. Many improvements in accuracy and repeatability have been achieved by designs using double-nested diaphragms and other clever innovations, all of which, of course, add to the cost. See the figure on page 5-22.

Electronic Sensors – In addition to the mechanical instruments, there are families of electronic pressure sensors. Some work on the same mechanical principles and others rely on strictly electronic response. Most of these devices, as mentioned above, convert their pressure data into a linear 4-20 mA signal for transmission to a control system outside the immediate area of the device. This eliminates the need for a separate signal converter or transmitter. Connection of these devices to a control network, or intrinsically safe system for hazardous locations, should be based on the same precautions as described in the section on temperature measurement (page 5-15) which will not be repeated here.

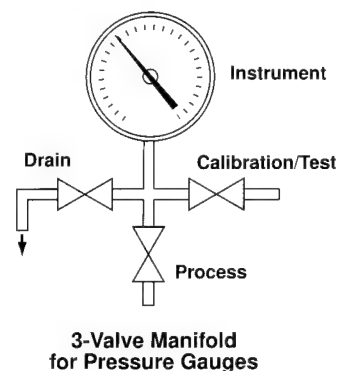
Strain Gauges – The original types of strain gauges worked by measuring the change in resistance of wires that were stretched as pressure increased. Modern strain gauges measure the deformation of a silicon chip or series of chips on a diaphragm, which is much more accurate ($\pm 0.25\%$) and reproducible. **Capacitance-type sensors** rely on the movement of a sensitive diaphragm to which a free-moving capacitor plate is attached. The capacitance of the circuit changes with the proximity of this plate to other energized system components. Capacitance is then converted to a more usable signal type. Many other specialized sensors exist, such as piezoelectric types, inductive magnetic types and optical transducers, but these are not as widely used.

Protective Devices – A number of conditions could damage pressure sensing instruments and should be considered for any installation. In cases where sudden wide pressure fluctuations are anticipated, a **pulsation dampener** is often used. These could take the form of the coil siphon tube commonly found in live steam service that permits condensation and keeps the vapors out of the sensing element, or a porous metal disk or orifice plate. These latter types may slow the response of the instrument by a few seconds, which should be taken into account in setting alarm set-points. **Freezing** can be prevented by using steam or electric-wire heat tracing or small internal electric heaters on larger instruments.

Most sensors are protected from **chemical attack** by means of protective diaphragms or seals. These can serve the added function of preventing process mixtures from entering and corroding or otherwise plugging the device. **Standard seals** usually use a corrosion resistant flexible diaphragm between the process and the sensing element. **Chemical seals** are devices that are filled with a non-compressible low-thermal expansion fluid, such as silicone oil, glycerin or low freezing solvents, to transmit the process pressure to the sensing element. They protect the element with no danger of process contamination in the event of a diaphragm leak or failure. Other protective devices employ **volumetric seals** (usually piston-type). The choice of seal should depend on the particular application and type of instrument.

Finally, good practice dictates that a **valve manifold** be incorporated into the design to enable safe draining and removal of pressure sensing instruments for maintenance or calibration, as shown in the figure at right.

Vacuum sensors are specialized pressure devices, also available in many types. Most common vacuum devices rely on the same principles as pressure gauges like Bourdon tubes and diaphragms. However, they cannot accurately read extremely high vacuum. For this purpose, liquid-filled manometers and McLeod gauges are used that can measure changes in vacuum of 0.01 torr or less. Other sophisticated designs capable of reading very high vacuums are also available. Your vacuum pump and system supplier can provide you with more detailed information.



Flow Measurement

Measuring the flowrate of liquids and gases is one of the most common determinations made in the CPI. Consequently, instruments for measuring it are available in a vast and sometimes confusing array of types, ranging from simple mechanical local-indicating meters to complex electronic sensors with built-in transmitters for remote connection. Flow totalizers, both mechanical and electronic, and flow switches are important extensions of the family. Most models are available for use in hazardous locations or intrinsically safe installations. Sanitary models are also widely available.

Most flow meters are mounted in-line in the process piping by flanged or threaded connections with the electronics (signal converter and transmitter) mounted integrally or positioned in a remote non-hazardous location. In order to ensure the stable flow pattern needed by most designs, many require a minimum length of straight-run pipe upstream and downstream of the meter or specify the inclusion of an upstream flow-straightener. A flow straightener is basically a section of straight pipe with a series of internal vanes or tubes designed to reduce the degree of turbulence.

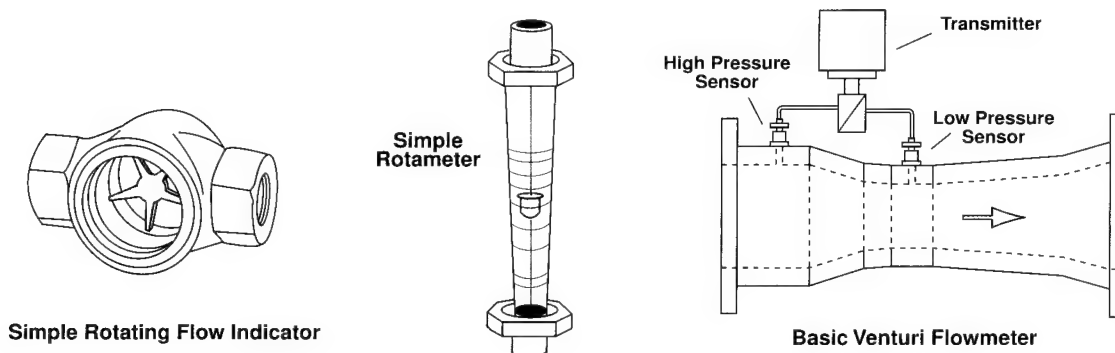
Meter Selection – Choosing a flowmeter can be a bit overwhelming, but the very first step is to understand why you need the meter and what you hope to accomplish with it. Remember that simple is best, and sometimes there is no more comforting feeling than to observe the rotation of a simple flow indicator (see figure below) to let you know that all is running well. If the application is a simple batch charging or processing operation, it may be possible to use the change in weight or volume of the source or receiving tank to determine flowrate.

Next, generate a list of process requirements, including types of fluids or materials to be measured, viscosity and density ranges, material compatibility issues, operating temperature limits, suspended solids, etc. Make a realistic determination of the accuracy you require, realizing that accuracies better than 1 or 2% are often unnecessary and add considerably to meter cost and limit your choices. Sometimes it is more important to have a meter that is reliable and gives consistent readings than one that is absolutely accurate. Will you read the meter locally or do you require a digital output as part of a control system? Consider your installation limitations and mounting position. If this is a meter requiring frequent removal or servicing, it may be necessary to include threaded unions or adopt a more expensive flanged design. Finally, always solicit the recommendations and comments of your supplier before making a purchase and carefully review the unit specifications, recommended maintenance schedule and spare parts list.

The major meter types and their characteristics are discussed below.

Rotameters – These devices, so named because of the spinning motion of the floats on many earlier models, are part of the class of devices known as *variable area meters*. They are a simple with very good accuracy, well-suited to many applications and capable of handling many fluid types, including corrosives and gases. See the diagram of a basic rotameter below. Rotameters are relatively easy to keep clean, except when the fluid contains fibrous suspended solids that can cling to the float. Manufacturers provide correction curves and performance characteristics so that the meter can be calibrated for fluids of various densities and viscosities.

Differential Pressure – These are one of the most widely used meters. They measure flow by comparing the pressures of a moving liquid before and after a restriction, such as an orifice plate, venturi tube or pipe elbow, based on the Bernoulli principle. They range from simple pitot tubes to modern electronic venturi meters, which employ a differential pressure sensor and a separate signal transmitter. They are suitable for many applications, but the measurable flow range is



relatively small and it is necessary to know the density of the liquid to convert pressure data into a flow value. In large installations, the pressure drop across the restriction can require an increase in pumping horsepower.

The relationship between pressure differential and flow is given by the following equation [64]:

$$\text{Flow} = k \sqrt{v^2 / 2gc}$$

where v = velocity and k is a constant unique to the meter. Since adherence to the above equation is never exact, full-range calibration is recommended. It is also usually important to observe certain minimum upstream straight-length requirements, to take steps to avoid the accumulation of gas or debris at the orifice and maintain a minimum necessary flowrate for accurate measurements.

The **venturi meter** (see the figure on the opposite page) is a particular type of differential flowmeter. The diameter typically converges at an angle of about 21° . At the point of constriction, fluid velocity increases and pressure drops proportionally to flowrate. This allows the placement of two pressure sensors and a differential pressure meter. These meters may usually be mounted in any position to suit installation needs, but do not work well when the Reynold's number for flow in the pipe is below 100,000.

Electromagnetic flow meters (or "magmeters") are based on the principle of electromagnetic induction and work by measuring the voltage induced in an electrically-conductive liquid moving through a fixed magnetic field. The magnetic field is produced by a winding outside the pipe, while the voltage, in the low millivolt range, is measured by small electrodes in the liquid path. The measurement is independent of density, viscosity and temperature, but the liquid being measured must be electrically conductive (greater than $20 \mu\text{-siemens/cm}$). All aqueous liquids and slurries fall into this category, but most pure organic solvents do not, and neither do gases. The meters are relatively simple, have no moving parts, generate no pressure drop and are accurate over a wide flow range of about 1:10. They are moderately expensive. A source of power (typically AC) and a transmitter are required. Depending on the design, valves are usually required on both sides to enable zero-flow calibration with the meter full. Electrode fouling is the primary concern with these otherwise rugged meters.

Turbine flow meters are one of the most common types used for low to medium viscosity liquids. They work by converting the speed of rotation of a turbine in the liquid stream to a digital output via a separate converter/transmitter. They are accurate with good flow range (~1:10) and can be used in high pressure or high temperature situations. These meters are relatively small and many turbine or impeller types are available; cost increases with accuracy. The major limitation may be that they are not suitable for liquids with suspended solids that could foul the moving elements.

Positive displacement meters can include many types such as sliding vane, rotary piston, oval gear nutating disk, and oscillating piston types. These meters are accurate and can provide a local mechanical readout without a power supply, as well as a signal for electronic conversion/transmission. They are not suitable for liquids with suspended solids, but work well on viscous liquids and are available for gas measurement. Similar to positive displacement pumps in design (see page 3-2), they work on the principle that the sensor moves a fixed distance per fixed volume of liquid, and require tight tolerances. Therefore they cannot handle slurries and suspended solids. Otherwise, they are accurate over a wide range and give very reproducible results.

Coriolis-type mass flow meters represent a departure from the types above in that they directly measure *mass* and not volumetric flow. This can be advantageous in many situations since density is not a factor. They are very accurate over wide ranges of flow (1:100). However, they are limited to relatively small sizes and moderate temperatures ($<200^\circ\text{C}$). They usually consist of a small-diameter tube or set of tubes through which a portion of the flow is diverted. It is the minute deformation of this tube that is electronically measured. Pressure drop through the measurement tube can reach up to 100 psi. This type of meter works well for viscous fluids and suspended solids. However, erosion and corrosion can be significant. The meters are widely used due to their versatility, wide range and the broad range of fluids that they can handle. There are designs that can handle gases, cryogenic liquids and pulsatile flow.

Vortex flowmeters work by measuring the frequency of vortices created in the fluid stream around a blunt body placed in the liquid path. They are widely used but only work at high Reynolds numbers (high flowrates). Other types that are now available include ultrasonic and thermal meters, which are used mostly for special applications.

Level Measurement

As with other instrumentation discussed in this chapter, there is a wide variety of choices for determining the level of liquid in a vessel, most of which fall into a few main categories. All of the methods mentioned below are used industrially, but many are most useful in large operations or vessels that offer no visual examination of the contents. These include armored sight indicators or level glasses. The potential dangers of using level devices consisting of a glass tube for vessels containing process fluids are obvious. The simple bubble tube method, in which the pressure of air necessary to generate bubbles out the bottom of a dip tube is carefully measured, is also common.

Visual Observation – Where visual observation is possible, many pilot vessels are manufactured with internal graduations that the operator can use for volume calibration. Even in the absence of such graduations, it is still very valuable to create a volume chart based on the major internal landmarks (baffle arms, temperature probe tip, etc.). Another alternative, useful for transparent glass vessels, is to graduate the vessel manually and mark the graduations on a piece of chemically-resistant tape that can be mounted to the outside surface. The use of a stainless steel or PTFE graduated dipstick is quite often seen, but common sense must dictate the limits and procedures for its safe use.

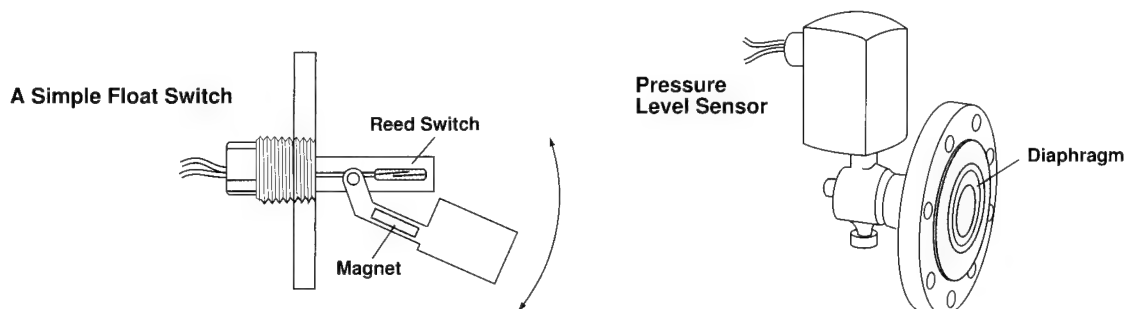
Surface Detection – Most small-scale pilot operations do not involve a great deal of automation and the types of manual measurements described above are acceptable. However, it may be wise to include discreet level detectors at critical points, for example, where an overfill would create a serious hazard. This reduces the problem of level measurement to one of surface level detection. Options include float switches, displacer switches, optical devices, and paddle sensors.

Weight – This is one of the most common methods for determining the mass, and thus the volume, in a vessel. Direct weight measurement by electronic load cells on which the vessel is mounted is widely used. Diaphragm-based differential pressure systems, with one sensor at the bottom of the vessel and one at the top, also work well, assuming they are calibrated for the specific liquid involved (see the figure below). Microprocessors makes possible “smart” level measurement systems, in which differential pressure data can be automatically corrected for temperature changes, tank shape and fluid characteristics. To calibrate any of these instruments, the density of the liquid must be known.

Other Electronic Methods – Other methods employ radar or lasers, directed from the top of the vessel down to the liquid surface, but these can give false readings if there is significant foaming. Another uses an ultrasonic sensor on the outside wall of the vessel, and therefore makes no contact with the process fluid. Other advanced electronic methods include microwaves, conductivity, proximity capacitance, impedance, radio frequency admittance, inductance and other characteristics. For more information on these methods see [159] or contact your equipment supplier.

Phase Boundary – A somewhat special application involves determining the level of the interface between two phases. This is accomplished by taking advantage of the differences of the two liquids in terms of electric or thermal conductivities, opacity, or sonic transmission. Ultrasonic probes, containing a built-in transmitter and receiver, are very handy for this purpose. Properly designed and weighted float switches can also serve this purpose. Other devices exist to accomplish this measurement, but again, in small pilot operations, if visual determination is possible, it is the easiest.

As always, if electronic instruments are used, pay particular care to their safety properties if they are to be used near flammable solvents or in locations classified as hazardous. Most instruments mentioned above are available in either explosion-proof or intrinsically safe configurations.

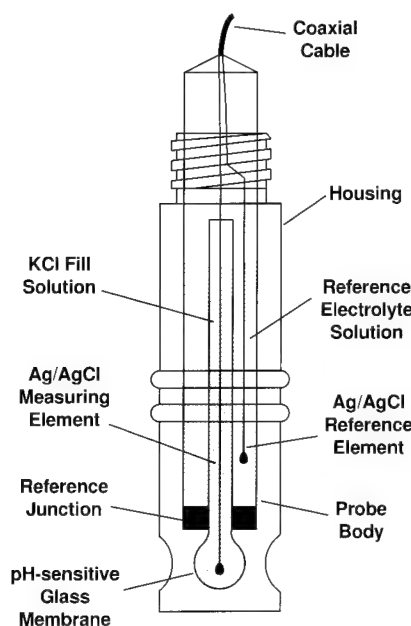


pH Measurement and Control

The measurement and control of pH is a very common industrial analysis but it is often poorly understood and frequently problematic. A review of some basic concepts and recommendations should prove helpful.

The acidity or alkalinity of an *aqueous* solution depends on the concentration of H^+ ions. The range of possible H^+ concentrations is very broad, ranging from greater than 1 mole/liter to less than 10^{-14} moles/liter. To make working with such a broad range more convenient, the pH scale was established. It is based on the negative \log_{10} of H^+ concentration, as shown in the table on page 5-24. For more information on the pH scale see "Buffers" on page 8-8.

pH sensors are extremely sensitive, capable of measuring changes of 0.01 pH units or less over a very broad range. In reality, achieving this accuracy reproducibly is not possible with most industrial pH probes. An accuracy of 0.2 pH units is more likely. This is partly due to the high sensitivity itself, which makes readings prone to the affects of electronic noise, electrode fouling, calibration drift, the change in probe response with temperature, and the true temperature dependence of pH itself. Adequate agitation and probe placement are also important in obtaining a correct pH reading.



Components of a Typical Industrial Combination pH Electrode

A pH measurement system consists of three components, the measurement electrode, the reference electrode and the pH meter. The **measurement electrode** consists of a specially-formulated glass membrane in the pH probe. The chamber is usually filled with a solution of KCl. Ions in the glass undergo an exchange with ions in the process liquid, generating an electric potential ranging on the order of ± 500 millivolts. The voltage potential is proportional to solution pH.

This millivolt signal is weak, and so must be read by a special high-impedance **meter** that amplifies the voltage signal and displays it as pH. The potential measured by a pH probe must always be compared to a **reference electrode**. In some cases, this is actually a second probe immersed in a standardized solution. But much more convenient are the combination electrodes that have the reference cell built right into the pH probe itself.

The reference electrode is often the most troublesome component of the system. It consists of a special silver or platinum wire element submerged in a filling solution inside the probe. The filling solution is in contact with the process liquid by means of a fritted or porous junction. If the filling solution is depleted, or the porous junction is plugged or not in full contact with the process liquid, an accurate reading is not possible. Long stabilization times for the probe can be one sign that the reference electrode is faulty. Likewise, if moving a hand towards or away from the probe results in a significant transitory change in the pH reading, it is also likely that there is something wrong with the reference electrode. The probe should be cleaned and refilled according to manufacturer's instructions. Common cleaning solutions include warm KCl solutions, ammonia (for clogged Ag/AgCl reference junctions), and urea, for dissolving proteins.

Standard pH probes are designed to operate within a temperature range from roughly 0 to 100°C and at pressures from full vacuum up to 100 psi. It is important to carefully assess the conditions for which the probe is specified before putting it into service. Never submerge an entire probe assembly unless it is an encapsulated unit designed for the purpose. Read any manufacturer's special care instructions completely.

Temperature Compensation – The response of glass pH electrodes varies with temperature. The further the temperature drifts from 0°C, and the farther from pH 7 the reading is, the greater the error will be. At 4 pH units from pH 7.0, a 10°C variation from zero results in an error of about 0.1 to 0.2 pH units. This may or may not be a problem depending on the required accuracy. Many pH meters include automatic temperature compensation circuitry, in which case a temperature probe must also be included in the loop. But manual temperature compensation is often misused. The proper way to read pH at temperatures other than 0°C is to first calibrate the pH probe in the proper buffers at the desired temperature, with the compensation dial adjusted to that temperature.

Calibration – Probes are typically calibrated using a two- or three-point method. First, the probe is allowed to equilibrate in a pH 7 buffer. The *gain* is then adjusted to read pH 7. Then the probe is placed in, say, a pH 4 or pH 10 buffer, and the *slope* is adjusted to read the correct pH. It is a good idea to recheck the pH 7 buffer and repeat the process if necessary.

Care of pH Probes – pH probes are highly specialized and often fragile instruments. Avoid rough handling to prevent breakage of the delicate glass bulb. In early pH technology, it was important that probes not be allowed to dry out in order to keep the outer glass layer hydrated. Newer probes will not be damaged by drying out, but it is often a good idea to soak the probe in a slightly acidic solution, which forces many contaminants out of the glass. Since most combination probes employ a AgCl reference solution, do not soak the probe in any solutions containing KCl, which can cause silver to precipitate. It is also not advisable to store probes in DI water as this can cause migration of the fill solution from the probe. When not in use, liquid filled combination probes should be cleaned with DI water, capped and stored dry. Consult the probe manufacturer for more detailed use and care instructions.

Common pH Control Problems – pH control may seem like a simple concept (add base to raise the pH, add acid to lower it) but in practice maintaining pH within the desired range is usually not that easy since so many things can affect it. Assuming that the probe is operating correctly and can be calibrated off-line and that there are no problems with the reference electrode (see above), and you've eliminated electrical noise/ground issues, then look at the system design.

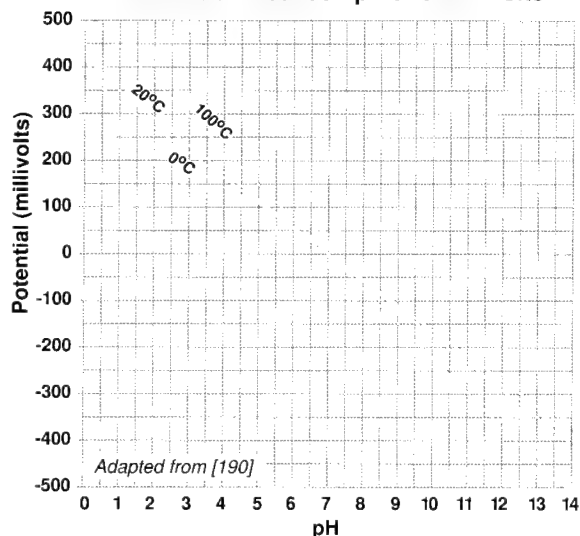
For example, take a look at the lag time in the response of acid/base pumps or valves, and the real-time titrant delivery rates. Consider the size of the control deadband and other control loop parameters (see page 5-26). Ensure that the concentration of the titrants is not too high, which can cause wild pH swings. If you are trying to maintain the pH of a moving stream, such as a waste effluent, it is critical to allow sufficient time for good mixing after acid/base addition, which may entail adding in-line static mixers or even an agitated holding vessel downstream of the addition port and upstream of the pH probe. If pH adjustment in an agitated vessel is the goal, ensure that agitation and turnover rates are sufficient in comparison to the rate of titrant addition. It may be necessary to add the acid/base to a separate closed circulation loop to ensure better distribution in very large tanks. It may simply be that the adjustment has to be undertaken slowly to allow sufficient mix time. pH control of slurries is particularly difficult.

The relative locations of the pH probe and titrant addition lines will also have a major effect on the operation of the control loop. Poor placement can cause long response delays or, worse, huge pH fluctuations. Try to determine the actual time required for a complete feedback cycle to be completed and compare this to the flowrates of all the streams involved. And finally, pay attention to the titration curve of the system you are trying to control. pH titration curves are *not* linear, and if you are trying to control pH on the steepest part of the curve, where small adjustments can mean major pH swings, it will only magnify your control problems. Consider the addition of a buffer, or using self-buffering titrants such as phosphoric acid or ammonia instead of HCl or NaOH. For more detailed information see [190].

The Negative-Logarithmic pH Scale

	H ⁺ Ion Concentration, moles/L			pH
		or		
Strong Acids	1	or	1×10^0	0
	0.1	or	1×10^{-1}	1
	0.01	or	1×10^{-2}	2
	0.001	or	1×10^{-3}	3
	0.0001	or	1×10^{-4}	4
	0.00001	or	1×10^{-5}	5
	0.000001	or	1×10^{-6}	6
	0.0000001	or	1×10^{-7}	7
	0.00000001	or	1×10^{-8}	8
	0.000000001	or	1×10^{-9}	9
Neutral	0.0000000001	or	1×10^{-10}	10
	0.00000000001	or	1×10^{-11}	11
	0.000000000001	or	1×10^{-12}	12
	0.0000000000001	or	1×10^{-13}	13
	0.00000000000001	or	1×10^{-14}	14
Strong Bases				

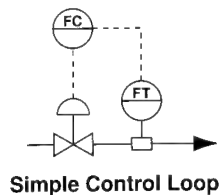
Correlation Between pH and Millivolts



Notes on Process Control

The field of process control is indeed a broad one, and it is beyond the scope of this book to delve into it in great detail. There are numerous excellent sources of information on the subject including [47, 191, 159]. Rather, we present some basic terminology and concepts that will enable readers to more easily research the subject themselves.

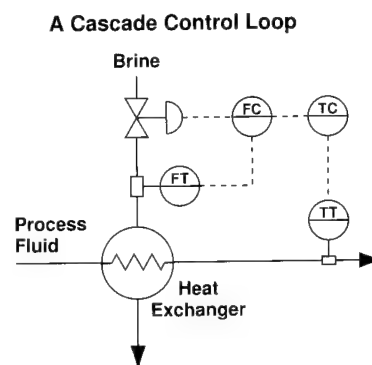
Basically, a controller is any device that operates automatically to regulate a variable. Examples include thermostats, which turn heating on or off to regulate temperature, pH controllers, which operate acid or base pumps to regulate pH of a process stream, or flow controllers, which automatically actuate valves to regulate fluid flow. Any control circuit must consist of at least three basic elements: a sensing element to “read” the process value, a control unit that makes the “decisions” about regulating the variable, and an actuation system to make the necessary adjustments as dictated by the control unit. In simplified control symbology, the diagram at the right illustrates a basic feedback control loop, in which a flow transmitter (FT) sends its signal to a flow controller (FC) that adjusts the position of a valve to maintain a flowrate setpoint.



In practice, most control systems are not so simple. It is common to have a dedicated, centrally located **programmable logic controller** (PLC) or PC-based control system, which receives process data from multiple field instruments and controls the actions of numerous feedback loops to regulate performance. The controller continually cycles through its sequence and makes adjustments every few seconds. The entire network is known as a distributed control system or DCS. Process variables received by the controller are usually analog, that is, continuously variable quantities, whereas outputs are more likely to be digital, or numerically discrete values.

The simplest type of control is “on/off”, in which an output is toggled on and off in an attempt to maintain a setpoint. On/off controllers make no distinction between large or small variations from the setpoint. An improvement on this is called “proportional control”, in which the controller adjusts its output in proportion to the variation from the setpoint. Thus, a heater need not be cycled on and off, but can be operated at, say, 50% power, giving smoother control and eliminating much of the cycling associated with on-off control. Other control algorithms include “integral control” in which the *rate of change* of the output is proportional to the input and “derivative control” in which output is proportional to the *rate of change of the input*. Many controllers actually use a combination of proportional, integral and derivative control to achieve the best results, and as such are known as **PID controllers**. They are found in many micro-processor-controlled systems and devices. The response time and accuracy of PID control loops are optimized by adjusting the mathematical coefficients associated with the control algorithms, a process called “tuning”. See “Understanding PID Controllers” on page 5-26.

Cascade control is an important technique in which the output from one control loop is used as the input to a second control loop. This may be best explained through an example. The figure at the right shows our simple flow control loop as part of a cascade control scheme. In this example, the system is “looking ahead”, as it were, to anticipate adjustments that must be made to the brine flowrate to maintain the proper temperature. Brine flowrate is secondary in importance to maintaining proper process temperature, and thus the flow controller becomes “slave” to the temperature controller from which it gets its setpoint. Cascade control, properly applied, can result in much smaller fluctuations in process variables and much more consistent control than even the best-tuned PID loops.



Setpoints that are programmed into controllers represent the desired process value. The controller will then strive to achieve and maintain that setpoint. Most controllers are also characterized by a **dead band**, which represents a span of values just above or just below the setpoint. The controller takes no action until the process value deviates outside the dead zone. This is necessary to prevent the controller from cycling on and off rapidly and continuously to correct inevitable minuscule variations. Nonetheless, the existence of the dead band can introduce significant **hysteresis**, also known as “lag time” or “overshoot”. Hysteresis causes the process variable to cyclically over- or under-shoot the setpoint, just as the temperature in a room fluctuates first above then below the thermostat setting.

Understanding PID Controllers

The control algorithm for PID controllers consists of three terms, as described below:

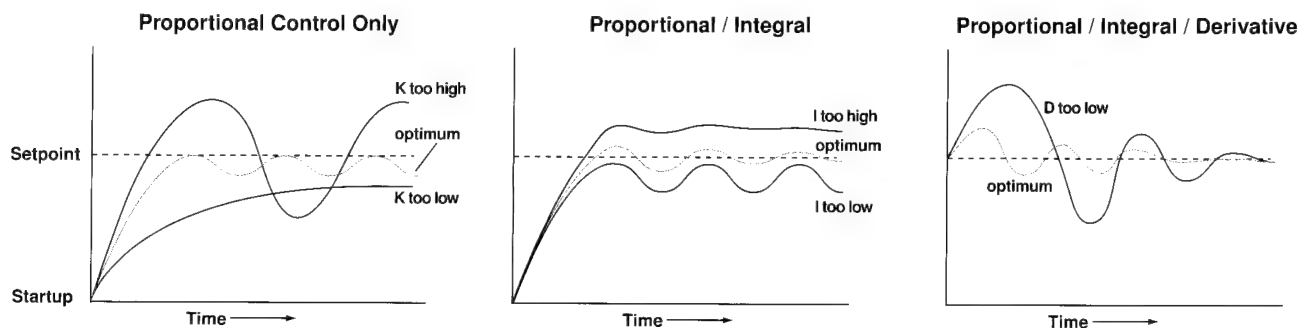
Proportional – In proportional control, the controller output is scaled back or increased in response to the size of the error or deviation from the process setpoint. The larger the deviation the greater the output. The mechanism usually involved is called “negative feedback” since, for example, heater output is *reduced* when temperature gets too high, and *increased* when too low. The opposite would result in the value spiralling out of control one way or the other. The proportionality constant between input and output is called the **gain**, here abbreviated as **K**. Let’s say when the process value deviates from the setpoint by 10% of full scale, the control output is 100%. In this case the gain has a value of 10. Another common term, the **proportional band width (PBW)** is the reciprocal of gain, or in this case 0.1. It means the same thing, that 10% variation results in 100% output change. If gain is set too high, the system oscillates considerably and even when properly tuned, steady-state will be achieved at a finite offset from the setpoint. See the figure below.

Integral – To overcome the offset problem associated with proportional control, this second term is added to the equation. Its function is to minimize deviation from setpoint at steady state by adjusting output based on the integral of setpoint error over time. The parameter **I**, called “integral action time” determines the relative influence of this term. High values (longer times) result in a slow approach to setpoint, smaller values or shorter times result in a faster approach. The optimum value of **I** results in minimum oscillations and reasonable time to reach steady state. Introducing this term, however, means that the proportional gain must be reduced for best results, which can mean slower overall response.

Derivative – Proportional/Integral (P/I) controllers can achieve good results in many situations, but when peak variations from setpoint are still higher than acceptable, the inclusion of the derivative term helps. This third component **R**, looks at the rate of change of setpoint error. Thus it makes no significant contribution at steady state, but comes into play if perturbations cause a sudden deviation. It minimizes rapid control movements which reduces oscillations. Therefore, an increased value of proportional gain can be used, improving overall system response. It is important that the three parameters **K**, **I** and **R**, be tuned in conjunction with each other to achieve the best results.

Tuning the Controller – Every application is different, and there is no set of tuning parameters (**K**, **I** and **R**) that will work in all situations. For proper operation, the parameters must be adjusted for each case. Most controllers have an auto-tune function that attempts to find the optimum values automatically by putting the system through a few (or many) cycles. However, this is not always available. Tuning a PID loop manually is more art than science. Trial-and-error is really the only way, but understanding how changes affect performance will help.

It is useful to first tune the proportional band width with the integral and derivative functions off (**I** and **D** set to zero). Start with a moderate value for **K** and go from there. In the figure below, note that even with **K** optimized, the average process value is offset from the setpoint. It is the role of the **D** function to minimize this offset. The second figure below gives an idea of its effects. As already mentioned, it is usually necessary to back off on **K** when the **I** function is turned on. **K** and **I** may work well together until there is a major perturbation, in which case process fluctuations may be higher than acceptable before steady state is reestablished. As shown in the third figure, increasing the value of the derivative parameter **D** helps to minimize these fluctuations. Thereafter it may be possible to increase the value of **K** again to speed start-ups. Typical values for **K** are between 0-100, for **I** between 0-10, and for **D**, between 0-2. Sources: [47, 159, 191].



6 Solvents

Contents

SOLVENT SAFETY

Notes on the Safe Handling of Solvents	6-2
NFPA Solvent Flammability Classifications	6-2
Relative Flammability Hazard of Selected Solvents	6-3

SOLVENT PROPERTIES AND SELECTION

Notes on Solvent Selection	6-4
Some Solvents Useful for Crystallization	6-4
Notes on Solvent Properties Table	6-5
Solvent Properties Table	6-6
Solubility Map of Common Solvents	6-16
Effect of Temperature on Specific Volume	6-17
Enthalpy of Vaporization vs. Boiling Point	6-18
Effect of Temperature on Enthalpy of Vaporization	6-19
Effect of Temperature on Viscosity	6-20
Effect of Temperature on Specific Heat	6-21

TEMPERATURE/VAPOR PRESSURE RELATIONSHIPS

Reduced Pressure Boiling Points for Some Common Solvents	6-22
Chart for Estimating Boiling Point vs. Pressure	6-23
Chart for Estimating Vapor Pressure at Elevated Temperatures	6-24

AZEOTROPES

Notes on Distillation and Azeotropes	6-25
Effect of Pressure on Azeotrope Composition	6-26
Water Azeotropes of Some Common Solvents	6-27
Binary Azeotropes of Some Common Solvents	6-28
Ternary Water-Containing Azeotropes of Some Common Solvents	6-40

MISCELLANEOUS

Solvents Limited for Pharmaceutical Use	6-44
Common Types of Denatured Ethanol	6-44

WATER DATA

Important Temperature Dependent Properties of Water	6-45
Common Water Purification Methods	6-47
Process Water Quality Specifications	6-47
Water Conductivity and Resistivity	6-48

One of the primary hazards associated with organic solvents is flammability. Solvent vapor/air mixtures are easily ignited by spark sources such as electric motors, thermostats, plugging in or unplugging equipment and static electric discharge. Electrostatic discharge sources can include clothing, plastics or other synthetic materials, moving or falling streams of liquids or powders, or the discharge of gases from high pressure cylinders. Nonpolar solvents such as heptane flowing through non-conducting hoses or tubes can build up significant static charges. The danger of electrostatic discharge increases at times of low humidity, such as in cold weather. At elevated temperatures, solvents generate more vapors and the danger of ignition is magnified. Many pure solvents burn with a nearly invisible flame making detection of a fire even more difficult. Also, the vapors of most flammable solvents are heavier than air and can accumulate in low places and travel along floors to ignition sources.

When transferring solvents, always ground and bond metal tanks and containers, and blanket vessels or containers with an inert gas such as nitrogen or argon. Argon has the advantage of being considerably heavier than air providing more efficient displacement of air. Periodically check the integrity of grounding cables and clamps and the continuity of grounded connections. Always keep containers tightly closed when not in use and use appropriate ventilation systems.

Many solvents, primarily ethers, can form explosive peroxides upon exposure to air and light. Store such solvents in a cool dark place and do not expose to air for prolonged periods of time. Never distill or attempt to dry ethers or other suspicious solvents without first testing for the presence of peroxides. Ethers are usually stabilized to prevent peroxide formation, and distillation removes the stabilizers. Distilled ethers should be re-stabilized.

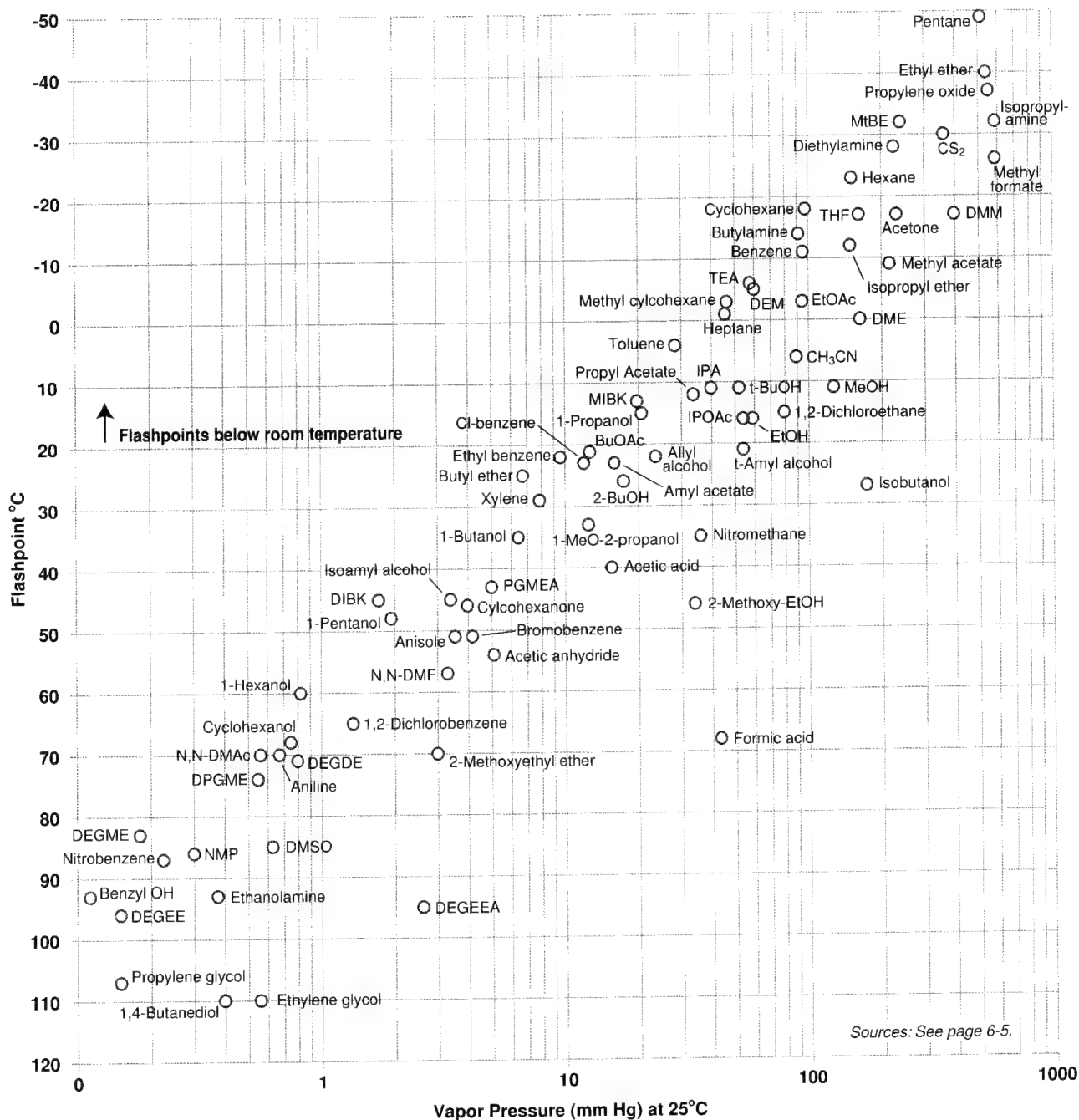
Many solvents, such as chlorinated hydrocarbons, are also highly toxic and their poisonous effects may be cumulative. Always avoid inhalation of vapors by using the proper personal protective equipment, such as organic-vapor or supplied-air respirators and ventilation systems. Particularly dangerous are benzene (a known carcinogen), aniline and nitrobenzene. Always read the MSDS for the solvents you are using to determine if a permissible exposure limit has been established by OSHA or another regulatory agency. This information, coupled with the vapor pressure, will give a relative idea of the potential for negative health effects.

Finally, always follow local guidelines and codes, which may limit the amounts of certain types of solvents that can be safely stored in a given location. These guidelines are usually based on the NFPA classification system which is summarized in the figure below. Solvents are classified according to the likelihood that they will ignite during use, which in turn is based on their flashpoints and boiling points. Solvents with lower boiling points have higher vapor pressures meaning that it is easier to develop a flammable concentration in air where the solvent is being used.

Boiling Point ↑	Flammable Liquids (Class I) (vapor pressure ≤ 40psia at 100 °F / 37.8 °C)		Combustible Liquids (Classes II and III)		
	IB	IC	II	IIIA	IIIB
	IA				
100 °F (37.8 °C)	73 °F (22.8 °C)	100 °F (37.8 °C)	140 °F (60 °C)	200 °F (93 °C)	
	Flashpoint →				

Source: [181]

Relative Flammability Hazard of Selected Solvents



This chart can be used as a general guide to solvent fire safety. Solvents are plotted according to their vapor pressures at 25°C and their flashpoints. Note that the flashpoint scale is inverted so that the closer a solvent is to the upper right hand corner (the higher the vapor pressure and the lower the flashpoint), the more hazardous it should be considered in terms of fire danger. Solvents with flashpoints at or below room temperature are indicated in the shaded region. Pentane, hexane and ethyl ether are now seldom used if a replacement solvent is available. Hexane is particularly prone to self-ignition because of its tendency to build up a static charge when flowing. Heptane is a slightly safer substitute as is MtBE for ethyl ether. To estimate vapor pressure at elevated temperatures, see the chart on page 6-24.

Notes on Solvent Selection

A solvent is a substance, usually liquid at room temperature, capable of dissolving another substance without reacting with it or chemically altering it. Solvents for chemistry are often classified according to their principle functionality or molecular structure. **Hydrocarbons** are organic solvents consisting of only hydrogen and carbon atoms, such as pentane or cyclohexane. They are usually very non-polar. **Halogenated** solvents contain at least one F, Cl, Br or I group, for example dichloromethane. **Protic** solvents are those capable of acting as hydrogen bond donors. These include polar solvents such as water, NH_3 and alcohols. Polar **aprotic**, or non-hydroxylic, solvents include DMSO, DMF and NMP.

Solvent selection for a given duty is usually a trade-off between safety, cost and effectiveness for the task. The latter would include effect on reaction rate, mixing characteristics, efficiency for extractions, the existence of favorable azeotropes, and ease of removal – both during work-up and from crystalline products during drying. When possible, use a single solvent rather than a mixture, to simplify charging and recovery. Try first to use inexpensive and readily available solvents. It is typical to select a short list of solvents based on reported properties, and then to narrow that field through experimental trials. Some key factors to consider are listed here:

Safety and Environmental Profile – This includes such factors as flashpoint, toxicity and the tendency to form explosive peroxides on degradation. Some solvents once widely used have been replaced by safer alternatives. For example, benzene and glyme, known to be carcinogenic, have been largely replaced as solvents by toluene and diethoxymethane, respectively. MtBE is a safer alternative for isopropyl ether since it does not tend to form peroxides. Consider also the costs associated with disposal and record keeping, particularly for EPA-listed compounds.

Polarity and Solubility Properties – Depending on the duty (reaction, extraction, crystallization, etc.) various considerations will apply. The relative solubility map on page 6-16 may be useful for comparisons.

Water Solubility – In organic chemistry, low water solubility can simplify phase separations, solvent drying operations and running anhydrous reactions. The ability to azeotropically remove water can also be a very useful characteristic.

Boiling and Freezing Points – This determines maximum reaction temperature and useful liquid range.

Reactivity – Some solvents slowly react under the proper conditions, affecting yield and causing the formation of impurities. The reactivity of many solvents, such as dichloromethane, is often not well-appreciated.

The tables and charts on the following pages will help the reader select a short list of solvents for further investigation based on the properties discussed above. This includes charts for estimating many properties vs. temperature. More information on selecting solvents can found in a number of excellent sources including [11, 70, 110, 205, 234].

Some Solvents Useful for Crystallization

The solvents listed below have been found to be successful, either alone or in combination, for the crystallization of organic compounds, although safety characteristics may make some unsuitable for large scale. Those marked with an asterisk* have been used, alone or in combination, for the resolution of chiral organic compounds by diastereomeric salt formation. Often the addition of only a small amount of a second solvent can drastically change the solvent properties of the primary solvent. Solvent-solute interactions are extremely complex and the presence of water and trace impurities can have a significant effect on yield and crystal properties. It is usually impossible to predict the results without some experimentation. See page 2-21 for more on developing successful crystallization processes.

Some Solvents Useful for Crystallizations

1 Water*	8 Isopropanol	15 THF
2 Acetic Acid	9 Methyl Acetate	16 Toluene
3 Acetonitrile*	10 Ethyl Acetate*	17 MIBK
4 Methanol*	11 Dichloromethane*	18 MEK
5 Ethanol*	12 n-Butanol	19 Hexane
6 Acetone*	13 Isobutanol	20 Cyclohexane
7 n-Propanol	14 t-Butanol	21 Heptane

*Useful for diastereomeric crystallizations

Sources: [70, 102, 145, 205]

Notes on the Solvent Properties Table

The table on pages 6-6 to 6-15 lists some useful physical properties of many common industrial solvents and organic liquids. The list includes those solvents most often encountered in the pharmaceutical and fine chemical industries, but is by no means intended to be comprehensive. More information can be obtained in the sources used in preparing the table: [8, 15, 19, 28, 35, 76, 78, 83, 95, 98, 152, 154, 166, 195, 205, 206, 225, 234].

The following data fields are reported in the table:

Common name

Synonyms or abbreviations – alternate names or acronyms in common use.

CAS # – a unique identifier recorded in the Chemical Abstract Services registry system.

Molecular structure

Chemical formula, written in the Hill order (C, then H, then other elements in alphabetical order).

Molecular weight – (also called relative molar mass) based on IUPAC standard atomic weights.

Density – density in gm/cm^3 at 20°C , 1 atmosphere absolute pressure (760 mm Hg).

Boiling point – boiling point in $^\circ\text{C}$. The temperature at which the vapor pressure equals 1 atmosphere absolute pressure (760 mm Hg, or 101.3 kPa).

Freezing point – freezing point in $^\circ\text{C}$ at 1 atmosphere absolute pressure (760 mm Hg) reported to the nearest $^\circ\text{C}$ (uncertainties are often greater than 2°C).

Refractive index – at 20°C , based on the sodium D line (589nm).

Solubility of the solvent in water – at 20°C , reported in weight percent. M = fully miscible; dec = decomposes.

Solubility of water in the solvent – at 20°C , reported in weight percent. M = fully miscible; dec = decomposes.

Dielectric constant – also called relative permittivity, at 20°C .

Hildebrand solubility parameter – reported in units of $\text{MPa}^{1/2}$. A function of molar latent heat of vaporization and specific volume, this is a measure of the amount of energy required to form a pocket and accommodate the transition state for the solute in the liquid matrix. It correlates roughly with the solubility of nonelectrolytes.

Coefficient of expansion – reported as a decimal, per $^\circ\text{C}$ of temperature change. Assuming linear expansion is a simplification, but a very good approximation over the normal liquid range of most solvents.

Enthalpy of vaporization – ΔH_{vap} at the normal boiling point in cal/g mole.

Specific heat – also called isobaric heat capacity, C_p , in cal/g mol $^\circ\text{C}$.

Flash point – flashpoint in $^\circ\text{C}$ at 1 atmosphere absolute pressure (760 mm Hg). The minimum temperature at which the vapor pressure is high enough to form a flammable vapor mixture with air at the liquid surface. Values here are reported based on the “closed-cup” method.

Viscosity – reported in centipoise (cP) at 25°C at 760 mm Hg. Note that to convert to kinematic viscosity, simply divided by the density (see conversion factors in Chapter 11).

Characteristics – lists the primary safety or handling characteristics according to RCRA and OSHA.

Considerable variation was found in the values reported in the literature for physical properties and especially for the compositions of azeotropic mixtures. In many cases, the data were simply not available in published form. The format of the following tables allows readers to fill in their own data to keep it handy. The empty lines at the end of the table may be used to keep track of data on particular solvents that are not listed in the table. Readers are further encouraged to notify us at the website www.pprbook.com if they are able to supply any data that are missing for inclusion in future editions, or to report any apparent errors.

Solvent Properties Table

Common Name	Synonyms, Abbreviations	CAS#	Structure	Formula	Mol. Wt.	Density g/cm ³ (20°C)	Boiling Pt. °C	Freez. Pt. °C
Acetic Acid	Ethanoic acid	64-19-7		C ₂ H ₄ O ₂	60.05	1.049	118	17
Acetic Anhydride	Acetyl oxide	108-24-7		C ₄ H ₆ O ₃	102.09	1.082	139	-73
Acetone	Dimethyl Ketone, 2-propanone	67-64-1		C ₃ H ₆ O	58.08	0.791	56	-94
Acetonitrile	Methyl cyanide	75-05-8		C ₂ H ₃ N	41.05	0.786	82	-46
Allyl Alcohol	2-Propen-1-ol	107-18-6		C ₃ H ₆ O	58.08	0.854	97	-129
Amyl Acetate	Pentyl acetate	628-63-7		C ₇ H ₁₄ O ₂	130.19	0.876	149	-100
t-Amyl Alcohol	2-Methyl-2-butanol, t-pentyl alcohol	75-85-4		C ₅ H ₁₂ O	88.15	0.805	102	-12
Aniline	Aminobenzene, phenylamine	62-53-3		C ₆ H ₇ N	93.13	1.022	184	-6
Anisole	Methoxy benzene, Methyl phenyl ether	100-66-3		C ₇ H ₈ O	108.14	0.995	154	-38
Benzaldehyde	Benzenecarboxaldehyde	100-52-7		C ₇ H ₆ O	106.12	1.044	178	-26
Benzene	Benzol	71-43-2		C ₆ H ₆	78.11	0.874	80	6
Benzyl Alcohol	Benzenemethanol	100-51-6		C ₇ H ₈ O	108.14	1.045	205	-15
Bromobenzene	Monobromobenzene, phenylbromide	108-86-1		C ₆ H ₅ Br	157.02	1.491	156	-31
1,3-Butanediol	1,3-Butylene glycol	107-88-0		C ₄ H ₁₀ O ₂	90.12	1.005	207	77
1,4-Butanediol	1,4-Butylene glycol	110-63-4		C ₄ H ₁₀ O ₂	90.12	1.017	230	18
1-Butanol	n-Butyl alcohol	71-36-3		C ₄ H ₁₀ O	74.12	0.810	118	-90
2-Butanol	sec-Butyl alcohol	78-92-2		C ₄ H ₁₀ O	74.12	0.808	98	-115
t-Butanol	2-Methyl-2-propanol	75-65-0		C ₄ H ₁₀ O	74.12	0.775	83	25
2-Butoxyethanol	Butyl cellosolve, E.G. monobutyl ether	111-76-2		C ₆ H ₁₄ O ₂	118.18	0.903	171	-75
Butyl Acetate	n-Butyl Acetate	123-86-4		C ₆ H ₁₂ O ₂	116.16	0.882	125	-78
Butylamine	1-Butanamine, MNBA	109-73-9		C ₄ H ₁₁ N	73.14	0.740	78	-49
Butyl Ether	Dibutyl ether	142-96-1		C ₈ H ₁₈ O	130.23	0.764	142	-98
Chlorobenzene	Monochlorobenzene, Dowtherm E	108-90-7		C ₆ H ₅ Cl	112.56	1.107	132	-45
Chloroform	Trichloromethane	67-66-3		CHCl ₃	119.38	1.492	61	-63
Cyclohexane	Hexamethylene	110-82-7		C ₆ H ₁₂	84.16	0.779	81	6

See Table Notes on Page 6-5.

Refractive Index (20°C)	Solubility in Water %w/w	Water Solub. in Solvent %w/w	Dielectric Constant (20°C)	Hildebrand Solubility Parameter	Exp. Coeff. / °C	ΔH_{vap} cal/g-mol (at NBP)	Specific Heat cal/mol/°C	Flash Point °C	Viscosity Cp (25°C)	Characteristics
1.3719	M	M	6.2	20.7		5801	29.4	40	1.15	Corrosive
1.3900	dec	dec	20.7	20.9	0.00145	9846	44.6	54	0.91	Corrosive, lachrymator
1.3590	M	M	20.7	20.5	0.00100	7076	23.8	-17	0.33	Flammable
1.3441	M	M	38.8	24.3	0.00140	7134	22.1	6	0.38	Toxic, corrosive
1.4120	M	M	21.6	24.3		9550	38.6	22	1.21	Highly toxic, flamm.
1.4020	0.2	0.9	4.8	16.6	0.00119	9764	60.4	23	43	Flammable
1.4050	9.0		5.8	17.8	0.00133	8233	66.4	21	3.7	Flammable, toxic
1.5860	3.8		6.9	22.8		11308	47.7	70	3.8	Highly toxic, suspected carcinogen
1.5160			4.3	18.4		8800	45.6	51	1.05	Irritant, hygroscopic
1.5450	0.003		17.9			10150	41.1	62	1.32	Suspected carcinogen, mutagen
1.5010	0.18	0.063	2.3	18.8	0.00138	7332	31.8	-11	0.65	Flammable, toxic, suspected carcinogen
1.5400	0.03		13.1	22.1		12068	58.4	93	0.06	Irritant, hygroscopic
1.5590	0.04		5.4	20.1		10158	36.3	51	1.1	Irritant
1.4400	M	M	28.8	25.6		13969		121	104	Irritant, hygroscopic
1.4450	M	M	31.0	24.7		13075	47.8	110	65	Irritant
1.3990	7.45	20.5	17.5	23.3	0.00094	10434	41.0	35	2.9	Flammable, toxic
1.3970	19.8	65.1	16.6	22.1	0.00091	9916	40.0	26	3.7	Flammable, irritant
1.3870	M	M	1.8	20.3		9317	52.6	11	3.4	Flammable, irritant
1.4190	M	M	9.3	19.4	0.00092	10384	55.9	60	6.4	Toxic, irritant
1.3940	0.5	1.6	5.0	17.6	0.00121	8584	53.4	22	0.73	Flammable, irritant
1.4010			4.9	17.9		7600	28.3	-14	0.68	Flammable, corrosive
1.3988	0.03		3.1	14.6		8724	48.8	25	0.65	Flammable, irritant
1.5240	0.049	0.003	5.6	19.4	0.00098	8814	35.0	23	0.8	Flammable, toxic
1.4460	0.82	0.2	4.8	19.0	0.00126	7021	27.4	none	0.57	Suspected carcinogen
1.4260	0.0055	0.01	2.2	16.8	0.00012	7140	37.0	-18	0.98	Flammable, toxic

Solvent Properties Table (continued)

Common Name	Synonyms, Abbreviations	CAS#	Structure	Formula	Mol. Wt.	Density g/cm ³ (20°C)	Boiling Pt. °C	Freez. Pt. °C
Cyclohexanol	Cyclohexyl alcohol, Hexalin	108-93-0		C ₆ H ₁₂ O	100.16	0.948	160	21
Cyclohexanone	Cyclohexyl ketone	108-94-1		C ₆ H ₁₀ O	98.15	0.947	155	-47
Cyclopentane	Pentamethylene	287-92-3		C ₅ H ₁₀	70.14	0.751	50	-94
1,2-Dichlorobenzene	o-Dichlorobenzol	95-50-1		C ₆ H ₄ Cl ₂	147	1.306	180	-17
1,2 Dichloroethane	Ethylene dichloride, DCE	107-06-2		C ₂ H ₄ Cl ₂	98.96	1.256	83	-35
Dichloromethane	Methylene chloride	75-09-2		CH ₂ Cl ₂	84.93	1.325	40	-97
Diethoxymethane	Diethyl formal, DEM	462-95-3		C ₅ H ₁₂ O ₂	104.15	0.839	87	-65
Diethylamine	DEA	109-89-7		C ₄ H ₁₁ N	73.14	0.707	55	-50
Diethylene Glycol	2,2-Oxydiethanol	111-46-6		C ₄ H ₁₀ O ₃	106.12	1.118	245	-10
Diethylene Glycol Diethyl Ether	2-Ethoxyethyl ether, Ethyl diglyme	112-36-7		C ₈ H ₁₈ O ₃	162.22	0.909	185	-64
Diethylene Glycol Ethyl Ether	2-(2-Ethoxyethoxy)-ethanol, Carbitol	111-90-0		C ₆ H ₁₄ O ₃	134.18	0.999	202	-55
Dieth. Glycol Ethyl Ether Acetate	2-(2-ethoxyethoxy)-ethyl acetate	112-15-2		C ₈ H ₁₆ O ₄	176.21	1.012	218	-25
Diethylene Glycol Hexyl Ether	2-(2-hexylethoxy)-ethanol, Hexyl Carbitol	112-59-4		C ₁₀ H ₂₂ O ₃	190.29	0.935	260	-40
Diethylene Glycol Methyl Ether	2-(2-Methoxyethoxy)-ethanol, Methyl Carbitol	111-77-3		C ₅ H ₁₂ O ₃	120.15	1.010	194	-70
Diisobutyl Ketone	2,6-Dimethyl-4-heptanone	108-83-8		C ₉ H ₁₈ O	142.24	0.808	169	-20.9
1,2-Dimethoxyethane	Glyme, Ethylene Glycol Dimethyl Ether, DME	110-71-4		C ₄ H ₁₀ O ₂	90.12	0.867	85	-58
Dimethoxymethane	Methylal, Methylene dimethyl ether, DMM	109-87-5		C ₃ H ₈ O ₂	76.10	0.860	41	-105
N,N-Dimethylacetimide	DMAC	127-19-5		C ₄ H ₉ NO	87.12	0.937	165	-20
Dimethyl Carbonate	Methyl Carbonate	616-38-6		C ₃ H ₆ O ₃	90.08	1.069	90	3
N,N-Dimethylformamide	DMF	68-12-2		C ₃ H ₇ NO	73.10	0.944	153	-98
Dimethyl Sulfide	Methyl sulfide	75-18-3		C ₂ H ₆ S	62.13	0.846	38	-98
Dimethyl Sulfoxide	Methyl Sulfoxide, DMSO	67-68-5		C ₂ H ₆ OS	78.13	1.101	189	19
1,4-Dioxane	p-Dioxane, 1,4-Diethylene dioxide	123-91-1		C ₄ H ₈ O ₂	88.11	1.034	101	12
Dipropylene Glycol	1,1'-Oxydi-2-propanol	25265-71-8		C ₆ H ₁₄ O ₃	134.18	1.023	230	-70

See Table Notes on Page 6-5.

Refractive Index (20°C)	Solubility in Water %w/w	Water Solub. in Solvent %w/w	Dielectric Constant (20°C)	Hildebrand Solubility Parameter	Exp. Coeff / °C	ΔH_{vap} cal/g-mol (at NBP)	Specific Heat cal/mol/°C	Flash Point °C	Viscosity Cp (25°C)	Characteristics
1.4650	4.3	11.8	15.0	23.3	0.00077	10900	50.0	68	54.5	Irritant, hygroscopic
1.4500	2.3	8	18.3	20.3	0.00090	9016	43.8	46	2.2	Corrosive, toxic
1.4000			2.0			6520	30.8	-37	0.41	Flammable, irritant
1.5510	<0.01	<0.01	9.9	18.8		9555	39.8	65	1.32	Toxic, irritant
1.4450	0.81	0.15	10.4	20.1	0.00116	7623	30.9	15	0.9	Suspected carcinogen, flammable
1.4240	1.3	0.2	8.9	19.8	0.00137	6715	23.8	none	0.44	Suspected carcinogen, toxic
1.3730	4.2	1.3	2.5	16.8		8350		-5	0.41	Flammable, irritant
1.3850	M	M	3.6	16.4		6656		-28	0.35	Flammable, corrosive
1.4460	M	M	31.7	26.4	0.00064	15918	57.3	143	35.7	Irritant, hygroscopic
1.4120	M	M	5.7	15.8		10500		71	1.4	Irritant
1.4270	M	M		20.1	0.00084	12886	73.0	96	4.5	Irritant, hygroscopic
1.4210	M	M		16.5	0.00106	11330	82.1	95	2.8	Irritant
1.4381	1.7	56.3			0.00084			140		Corrosive
1.4260	M	M		19.6	0.00088	10870	64.9	83	3.9	Irritant
1.4130	0.05	0.7		14.9	0.00102	9458	70.8	45	1	Irritant
1.3790	M	M		16.4		6700	39.5	0	1.1	Flammable, possible teratogen
1.3540	33		2.7				38.7	-17	0.33	Flammable, irritant
1.4380	M	M	37.8	21.7		10360		70	0.92	Irritant
1.3680			3.1	20.3		8299		18		Flammable, moisture sensitive
1.4310	M	M	36.7	24.8	0.00096	10074	35.5	57	0.82	Teratogen, irritant
1.4360			6.7			6450	54.3	-36	0.28	Flammable, stench
1.4790	M	M	46.7	26.6	0.00088	12636	36.5	85	1.99	Irritant, hygroscopic
1.4220	M	M	2.2	20.5	0.00120	8510	35.6	12	1.30	Suspected carcinogen, flammable, peroxides
1.4410	M	M		16.8	0.00070	8715	38.7	137	75.0	Irritant

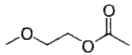
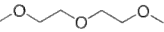








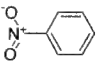

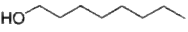




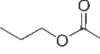
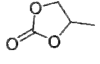
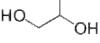
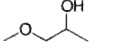
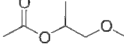


Solvent Properties Table (continued)

Common Name	Synonyms, Abbreviations	CAS#	Structure	Formula	Mol. Wt.	Density g/cm ³ (20°C)	Boiling Pt.°C	Freez. Pt.°C
Dipropylene Glycol Methyl Ether	Dowanol 50B	34590-94-8		C ₇ H ₁₆ O ₃	148.20	0.951	90	-112
Ethanol	Ethyl alcohol	64-17-5		C ₂ H ₆ O	46.07	0.794	78	-114
Ethanolamine	2-Aminoethanol	141-43-5		C ₂ H ₇ NO	61.08	1.012	170	11
2-Ethoxyethanol	Cellosolve, Ethylene glycol monoethyl ether	110-80-5		C ₄ H ₁₀ O ₂	90.12	0.930	135	-90
Ethyl Acetate	EtOAc	141-78-6		C ₄ H ₈ O ₂	88.11	0.902	77	-84
Ethylbenzene	Phenylethane	100-41-4		C ₈ H ₁₀	106.17	0.867	136	-95
Ethylene Glycol	1,2-Ethanediol	107-21-1		C ₂ H ₆ O ₂	62.07	1.113	197	-13
Ethylene Glycol Diethyl Ether	1,2-Diethoxyethane, ethyl glyme	629-14-1		C ₆ H ₁₄ O ₂	118.18	0.842	121	-74
Ethyl Ether	Ether, diethyl ether	60-29-7		C ₄ H ₁₀ O	74.12	0.706	35	-116
Formamide	Methanamide	75-12-7		CH ₃ NO	45.04	1.134	210	2
Formic Acid	Methanoic acid	64-18-6		CH ₂ O ₂	46.03	1.220	100	8.3
Furfural	2-Furaldehyde	98-01-1		C ₅ H ₄ O ₂	96.09	1.160	162	-37
Glycerol	Glycerine	56-81-5		C ₃ H ₈ O ₃	92.09	1.261	290	20
n-Heptane	Heptane	142-82-5		C ₇ H ₁₆	100.21	0.684	98	-91
n-Hexane	Hexane	110-54-3		C ₆ H ₁₄	86.18	0.659	69	-95
1-Hexanol	n-Hexyl alcohol	111-27-3		C ₆ H ₁₄ O	102.18	0.814	156	-52
Hexylene Glycol	2-Methyl-2,4-pentanediol	107-41-5		C ₆ H ₁₄ O ₂	118.18	0.925	197	-40
Isoamyl Alcohol	3-Methyl-1-butanol	123-51-3		C ₅ H ₁₂ O	88.15	0.809	130	-117
Isobutanol	Isobutyl alcohol, 2-Methyl-1-propanol	78-83-1		C ₄ H ₁₀ O	74.12	0.803	108	-108
Isobutyl Acetate	Acetic acid isobutyl ester, IBuOAc	110-19-0		C ₆ H ₁₂ O	116.16	0.868	117	-99
Isopropanol	2-Propanol, Lactol, IPA	67-63-0		C ₃ H ₈ O	60.10	0.785	82	-90
Isopropyl Acetate	IPAC	108-21-4		C ₅ H ₁₀ O	102.13	0.872	87	-73
Isopropylamine	Monoisopropylamine	75-31-0		C ₃ H ₉ N	59.11	0.694	33	-95
Isopropyl Ether	Diisopropyl ether	108-20-3		C ₆ H ₁₄ O	102.18	0.725	68	-86
Methanol	Methyl alcohol	67-56-1		CH ₄ O	32.04	0.791	65	-98
2-Methoxyethanol	Methyl cellosolve, Eth. glycol methyl ether	109-86-4		C ₃ H ₈ O ₂	76.10	0.965	124	-85

See Table Notes on Page 6-5.

Refractive Index (20°C)	Solubility in Water %w/w	Water Solub. in Solvent %w/w	Dielectric Constant (20°C)	Hildebrand Solubility Parameter	Exp. Coeff / °C	ΔH_{vap} cal/g-mol (at NBP)	Specific Heat cal/mol/°C	Flash Point °C	Viscosity Cp (25°C)	Characteristics
1.4220	M	M		16.0	0.00094	9443	80.0	74	3.70	
1.3600	M	M	24.6	27.4	0.00110	9200	27.1	16	1.20	Toxic, flammable
1.4540	M	M	37.7	29.1	0.00079	12159	39.3	93	3.40	Corrosive, hygroscopic
1.4070	M	M	29.6	20.3	0.00097	9630	50.0	44	2.05	Teratogen, irritant
1.3720	7.7	3.3	6.0	18.6	0.00140	7744	40.4	-3	0.46	Flammable, toxic
1.4950	0.02	0.03	2.4	17.0	0.00090	8480	43.4	22	0.72	Flammable, irritant
1.4310	M	M	37.7	35.0	0.00070	12524	34.8	>110	20.9	Toxic, irritant
1.3923	20.4	3.3	3.9			8670	61.9	20	0.70	Flammable, irritant
1.3530	6.9	1.3	4.3	15.1	0.00110	6216	41.3	-40	0.24	Highly flamm., toxic
1.4470	M	M	84.0	43.4		18016	24.7	154	3.80	Teratogen, irritant
1.3704	M	M	58.5	24.7		5501	23.8	68	1.70	Corrosive, toxic
1.5260	8.3		42.1			10320	39.0	58	1.59	Corrosive, toxic
1.4740	M	M	42.5	32.1		18169	53.1	160	945	Irritant, hygroscopic
1.3870	0.005	0.005	1.9	15.3	0.00090	7645	50.7	-1	0.41	Highly flamm., irritant
1.3750	0.001	0.011	1.9	14.9	0.00130	6880	42.0	-23	0.31	Flammable, electrostatic buildup
1.4180	0.58	7.2	13.3	20.1		12078	56.6	60	5.40	Irritant
1.4270	M	M		20.1		12290	52.1	93		Irritant, hygroscopic
1.4060	2.0		14.7	21.5		12000	48.6	45	3.90	Irritant
1.3960	8.7	15	17.9	21.9	0.00095	10220	53.0	27	3.90	Flammable, toxic
1.3900	0.6	1.6	5.0	16.6	0.00013	8758	53.3	21	0.70	Flammable, irritant
1.3770	M	M	19.9	23.5	0.00105	9540	36.6	11	2.0	Flammable, irritant
1.3770	2.9	1.8		17.0	0.00131	8109	47.0	16	0.60	Flammable, irritant
1.3740	M	M	5.5	17.9		6501	37.8	-32	0.34	Flammable, corrosive
1.3680	1.2	0.62	3.9	14.1	0.00108	6936	51.7	-12	0.33	Flammable, irritant
1.3290	M	M	32.7	29.7	0.00120	8426	19.5	11	0.60	Flammable, toxic
1.4020	M	M	16.9	22.1	0.00095	10260	40.6	46	1.72	Teratogen, irritant



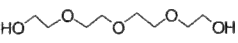



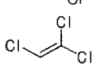

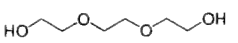
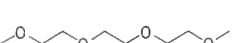

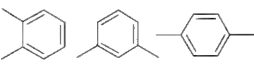
Solvent Properties Table (continued)

Common Name	Synonyms, Abbreviations	CAS#	Structure	Formula	Mol. Wt.	Density g/cm ³ (20°C)	Boiling Pt. °C	Freez. Pt. °C
2-Methoxyethyl Acetate	Methyl cellosolve acetate	110-49-6		C ₅ H ₁₀ O ₃	118.13	1.009	145	-65
2-Methoxyethyl Ether	Diglyme, Diethylene glycol dimethyl ether	111-96-6		C ₆ H ₁₄ O ₃	134.18	0.937	162	-64
Methyl Acetate	Acetic acid methyl ester	79-20-9		C ₃ H ₆ O	74.08	0.932	57.5	-98
Methyl t-Butyl Ether	t-Butyl methyl ether, MtBE	1634-04-4		C ₅ H ₁₂ O	88.15	0.740	55	-109
Methyl Cyclohexane	Cyclohexylmethane	108-87-2		C ₇ H ₁₄	98.19	0.770	101	-126
Methyl Ethyl Ketone	2-Butanone, MEK	78-93-3		C ₄ H ₈ O	72.11	0.805	80	-87
Methyl Formate	Methyl methanoate	107-31-3		C ₂ H ₄ O ₂	60.05	0.974	34	-100
Methyl Iodide	Iodomethane	74-88-4		CH ₃ I	141.94	2.280	42	-64
Methyl Isobutyl Ketone	4-Methyl-2-pentanone, Hexone, MIBK	108-10-1		C ₆ H ₁₂ O	100.16	0.801	117	-80
N-Methyl Pyrrolidone	1-Methyl-2-pyrrolidone, NMP	872-50-4		C ₅ H ₉ NO	99.13	1.028	202	-24
Nitrobenzene	Nitrobenzol, oil of mirbane	98-95-3		C ₆ H ₅ NO ₂	123.11	1.196	210	6
Nitromethane	Nitrocarbinol	75-52-5		CH ₃ NO ₂	61.04	1.127	101	-29
1-Octanol	Octyl alcohol, Capryl alcohol	111-87-5		C ₈ H ₁₈ O	130.23	0.827	196	-15
n-Pentane	Pentane	109-66-0		C ₅ H ₁₂	72.15	0.626	36	-130
1-Pentanol	n-Amyl alcohol	71-41-0		C ₅ H ₁₂ O	88.15	0.811	137	-78
3-Pentanone	Diethyl ketone	96-22-0		C ₅ H ₁₀ O	86.13	0.813	102	-40
1-Propanol	n-Propyl alcohol	71-23-8		C ₃ H ₈ O	60.10	0.804	97	-127
Propyl Acetate	n-Propyl acetate	109-60-4		C ₅ H ₁₀ O	102.13	0.888	102	-95
Propylene Carbonate	1,2-Propanediol cyclic carbonate	108-32-7		C ₃ H ₆ O ₃	102.09	1.189	240	-55
Propylene Glycol	1,2-Propanediol	57-55-6		C ₃ H ₈ O ₂	76.10	1.036	187	-60
Propylene Glycol Methyl Ether	1-Methoxy-2-propanol	107-98-2		C ₄ H ₁₀ O ₂	90.12	0.962	118	-142
Prop. Glycol Methyl Ether Acetate	1-Methoxy-2-propanol acetate	108-66-6		C ₆ H ₁₂ O ₂	132.16	0.968	145	
Propylene Oxide	1,2-Epoxypropane	75-56-9		C ₃ H ₆ O	58.08	0.830	34	-112
Pyridine	Azine	110-86-1		C ₅ H ₅ N	79.10	0.978	115	-42

See Table Notes on Page 6-5.

Refractive Index (20°C)	Solubility in Water %w/w	Water Solub. in Solvent %w/w	Dielectric Constant (20°C)	Hildebrand Solubility Parameter	Exp. Coeff / °C	ΔH_{vap} cal/g-mol (at NBP)	Specific Heat cal/mol/°C	Flash Point °C	Viscosity Cp (25°C)	Characteristics
1.4020	M	M	8.3	18.6		9638		43		Teratogen
1.4080	M	M	7.2	17.2		10000	54.1	70	2.0	Hygroscopic
1.3610	24.5	8.2	6.7	19.6	0.00140	7178	36.9	-9	0.37	Flammable, irritant
1.3690	4.3	1.4	2.6	15.5	0.00078	6893	44.8	-32	0.35	Flammable, irritant
1.4220			2.0	15.7		7481	44.2	-3	0.68	Flammable, irritant
1.3790	26	12	18.5	19.0	0.00100	7848	35.9	-3	0.41	Flammable, toxic
1.3430	33		8.5	21.5		6742	29.6	-26	0.34	Flammable, irritant
1.5310	1.0		7.0			6520	30.1	none	0.47	Suspected carcinogen, toxic, corrosive
1.3960	1.9	1.55	13.1	17.2	0.00094	8500	47.0	13	0.61	Flammable, toxic
1.4700	M	M	32.0	22.5	0.00080	12600	39.6	86	1.80	Irritant, hygroscopic
1.5510	0.19		35.7	22.2		12168	43.1	87	1.86	Highly toxic, irritant
1.3820	M	M	35.9	25.1		8124		35	0.65	Flammable
1.4290	0.6		10.3	17.3		11209	77.7	81	8.9	Irritant
1.3580	0.004	0.012	1.8	14.3	0.00095	6120	40.3	-49	0.24	Highly flammable
1.4090	2.6	9.5	13.9	20.3	0.00092	10630	62.8	48	4.0	Toxic, irritant
1.3920	5.0		17.0	17.8		8007	45.6	6	0.44	Flammable, irritant
1.3840	M	M	20.3	24.3	0.00096	9780	34.2	15	1.72	Flammable, irritant
1.3840	2.3	2.6	6.0	17.2		8165		12	0.56	Flammable, irritant
1.4210	21	8.3	64.0	27.2	0.00084	15182		132	2.4	Irritant
1.4320	M	M	32.0	27.0	0.00072	12853	45.1	107	56	Hygroscopic
1.4030	M	M		20.7		9640	52.2	33	1.80	Flammable, irritant
1.4020	19.8	5.9		17.0	0.00113	9414	55.5	43	1.1	Irritant
1.3660	40.5	12.8		17.4	0.00157	5157		-37	0.4	Suspected carcinogen, flammable
1.5100	M	M	12.4	21.9	0.00070	8374	33.6	20	0.88	Flammable, toxic

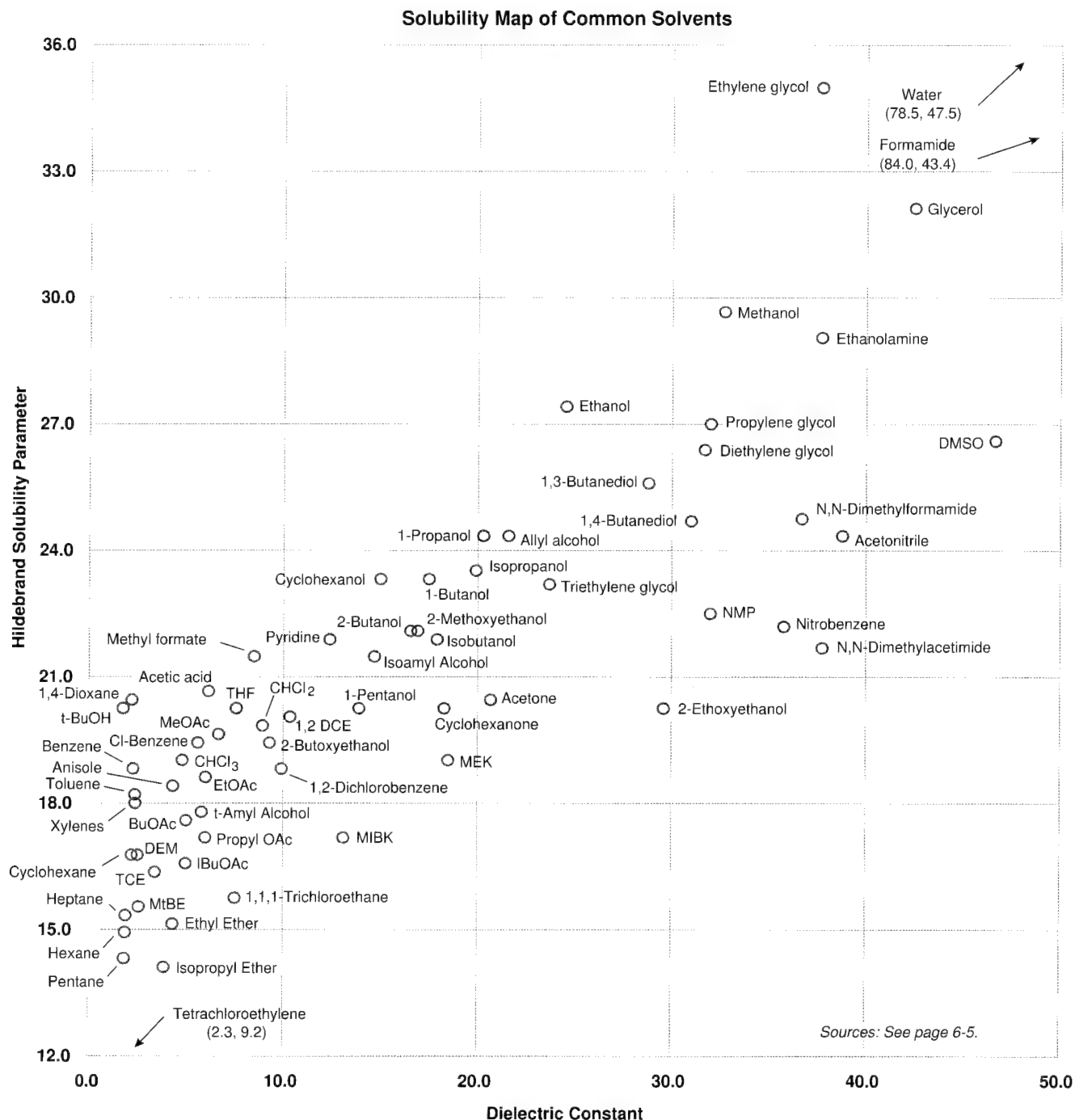
Solvent Properties Table (concluded)

Common Name	Synonyms, Abbreviations	CAS#	Structure	Formula	Mol. Wt.	Density g/cm ³ (20°C)	Boiling Pt. °C	Freez. Pt. °C
2-Pyrrolidone	2-Pyrrolidinone	616-45-5		C ₄ H ₇ N	85.11	1.120	245	24
Tetrachloroethylene	Perchloroethylene, Perk	127-18-4		C ₂ Cl ₄	165.83	1.623	121	-22
Tetraethylene Glycol		112-60-7		C ₈ H ₁₈ O ₅	194.23	1.125	314	-6
Tetrahydrofuran	THF, 1,4-Epoxybutane	109-99-9		C ₄ H ₈ O	72.11	0.889	66	-108
Toluene	Toluol, methylbenzene	108-88-3		C ₇ H ₈	92.14	0.865	110	-93
1,1,1-Trichloroethane	Methylchloroform	71-55-6		C ₂ H ₃ Cl ₃	133.41	1.338	75	-35
Trichloroethylene	Triclene, TCE	79-01-6		C ₂ HCl ₃	131.39	1.463	87	-85
Triethylamine	TEA	121-44-8		C ₆ H ₁₅ N	101.19	0.726	88.8	-115
Triethylene Glycol		112-27-6		C ₆ H ₁₄ O ₄	150.17	1.125	285	-7
Triethylene Glycol Dimethyl Ether	Triglyme, TGDME	112-49-2		C ₈ H ₁₈ O ₄	178.23	0.986	216	-45
Water		7732-18-5		H ₂ O	18.02	0.998	100	0
Xylenes	Xylol, Dimethylbenzene	1330-20-7		C ₈ H ₁₀	106.17	0.860	~140	~ -50

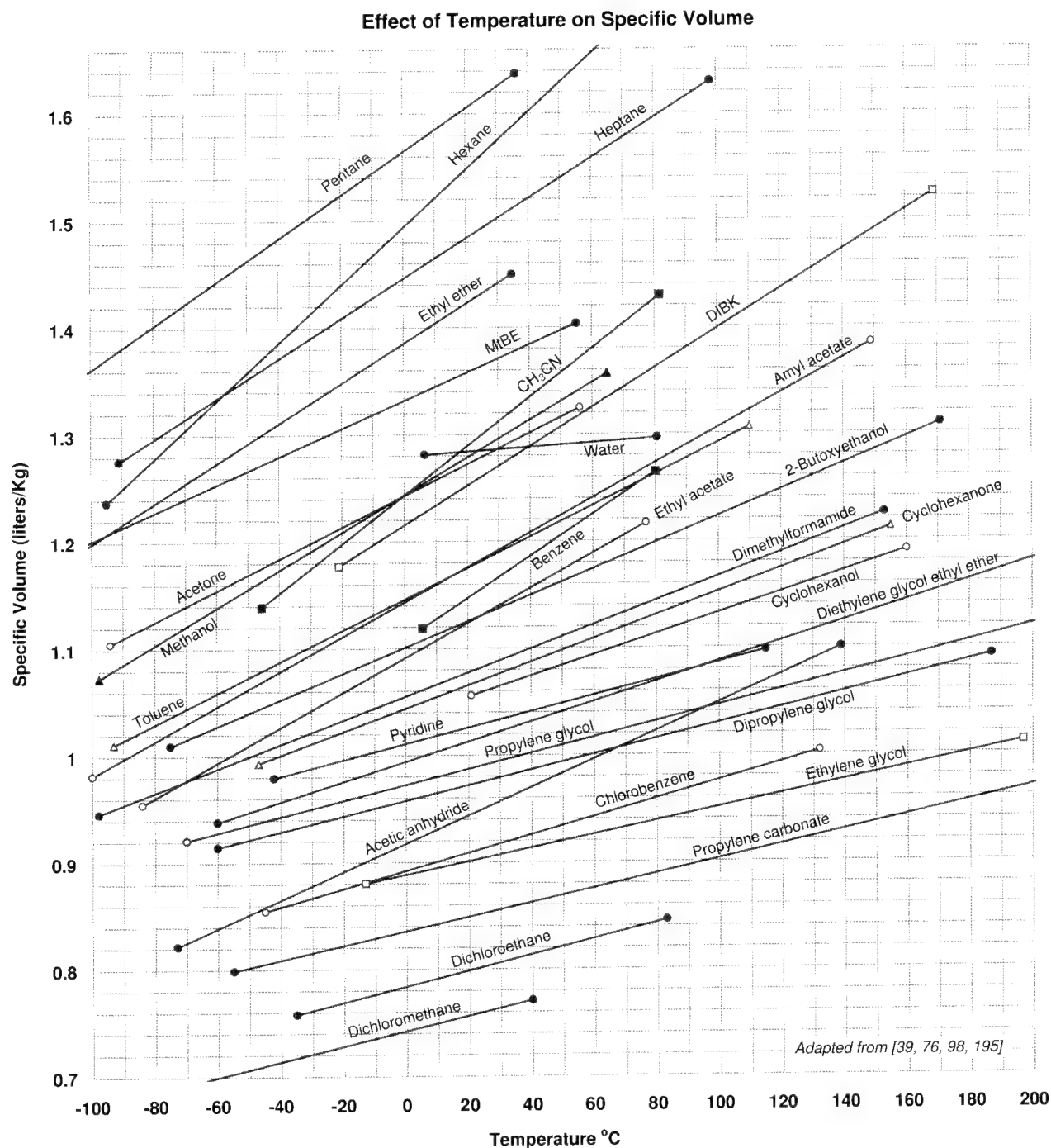
See Table Notes on Page 6-5.

Use the spaces above to record data for solvents not listed. Visit www.pprbook.com to report errors or to suggest additional data for future editions.

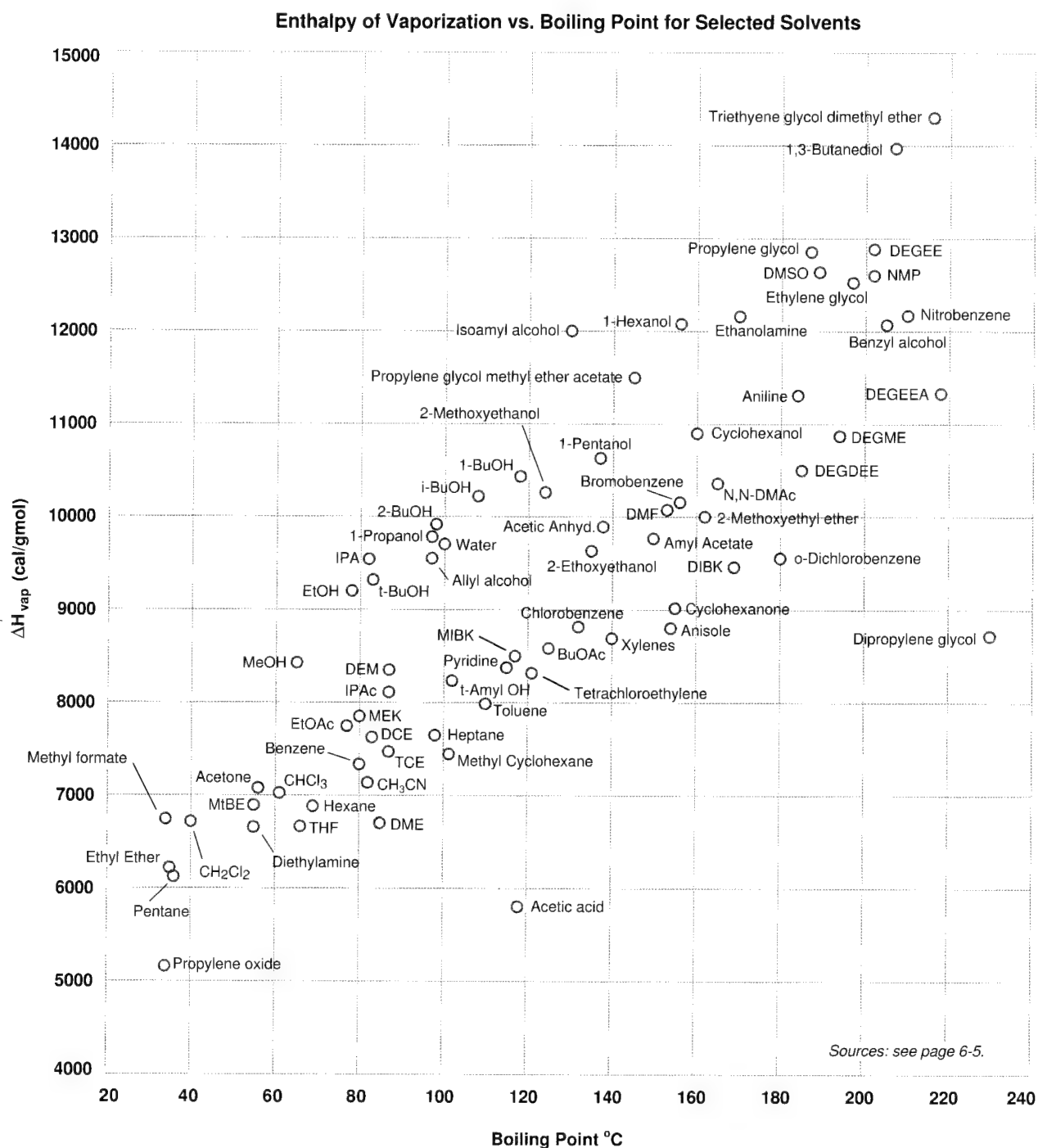
Refractive Index (20°C)	Solubility in Water %w/w	Water Solub. in Solvent %w/w	Dielectric Constant (20°C)	Hildebrand Solubility Parameter	Exp. Coeff / °C	ΔH_{vap} cal/g-mol (at NBP)	Specific Heat cal/mol/°C	Flash Point °C	Viscosity Cp (25°C)	Characteristics
1.4870	M	M						>110	13.3	Highly toxic, irritant
1.5060	0.015	0.008	2.3	18.5	0.00102	8316	34.9	none	0.88	Suspected carcinogen, mutagen
1.4590	M	M	20.4	22.7		21297	101.0	176	50	Irritant
1.4070	M	M	7.6	20.3	0.00110	6664	31.3	-17	0.55	Flammable, toxic
1.4960	0.06	0.04	2.4	18.2	0.00110	7985	41.0	4	0.59	Flammable, toxic
1.4366	0.13	0.05	7.5	15.8	0.00130	7780	31.9	none	0.65	Toxic, hygroscopic
1.4760	0.11	0.033	3.4	16.4	0.00117	7467	30.1	none	0.57	Suspected carcinogen, mutagen
1.4000	M	M	2.4	14.9		7409	38.5	-6	0.39	Flammable, corrosive
1.4550	M	M	23.7	23.2	0.00069	17097	78.8	165	47.8	Irritant, hygroscopic
1.4230	M	M	7.6	18.2		14300	75.6	110	4.1	Teratogen
1.3330	-	-	80.4	47.5	0.00021	9703	17.9	none	0.89	
1.4970	0.02	0.05	2.4	18.0	0.00100	8692	42.4	29	0.7	Flammable, toxic



This chart can be helpful when selecting a short list of solvents with similar solubility properties for a given duty or when seeking substitute solvents with improved safety or environmental profiles. Generally speaking, the higher the dielectric constant, the better the solvent will be for inorganic salts and polar compounds. The Hildebrand solubility parameter is a complex function that correlates roughly with the solubility of nonpolar organics. Also, the closer two solvents are in terms of the Hildebrand parameter, the more miscible they are likely to be. For more information on the aqueous solubility of various compounds, see page 8-11.

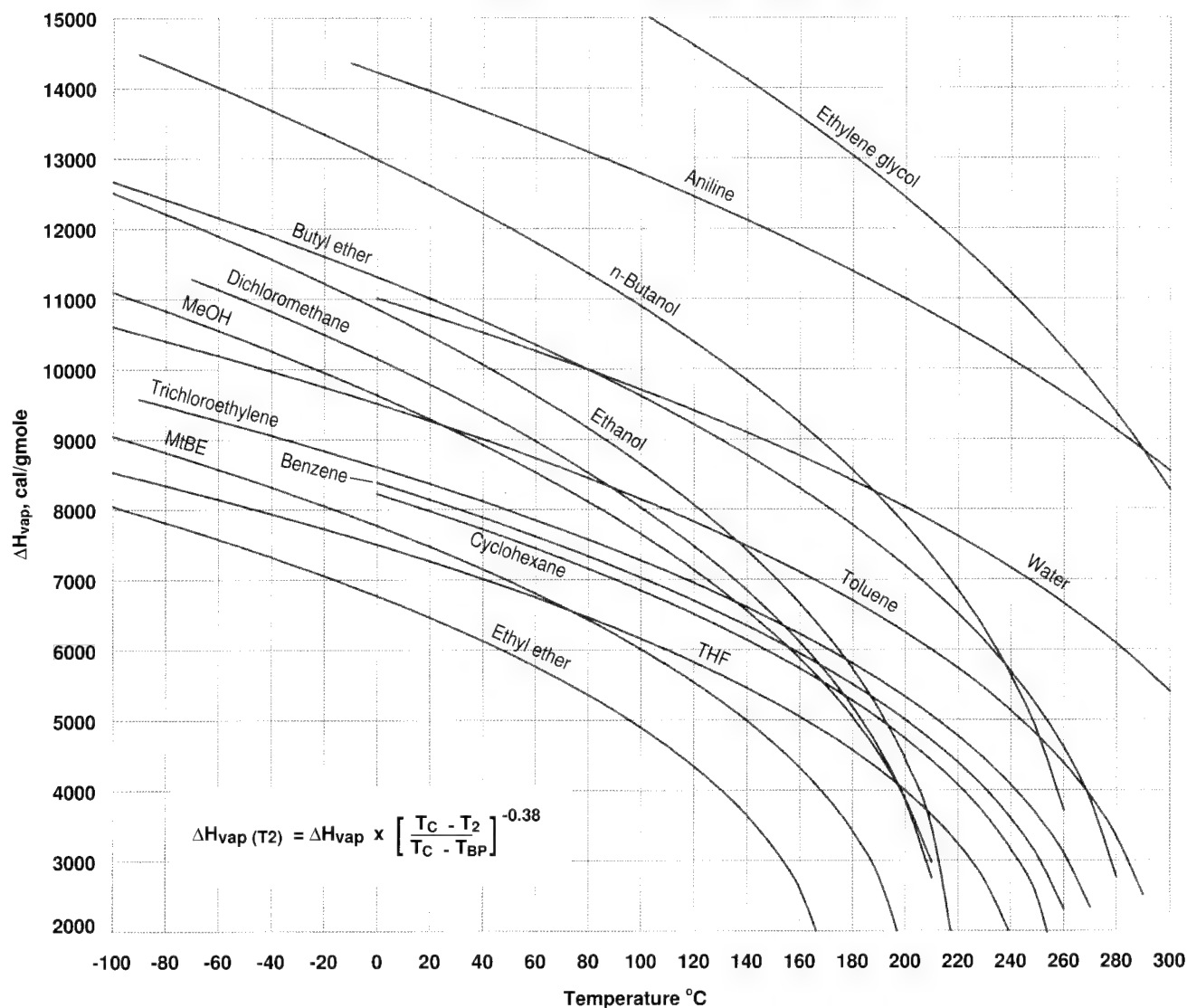


This chart illustrates the change in specific volume vs. temperature for some common solvents over their normal liquid ranges. It was prepared based on the assumption that the cubical expansion coefficient is linear over this range, which proves to be an excellent approximation. The endpoints of the lines represent the normal freezing and boiling points of each solvent. Note that, with the exception of water, most solvents increase in volume about 1% for each 10°C rise in temperature. This can result in significant volume variation, up to nearly 30% in some cases, and cannot be ignored when planning operations at the pilot or larger scale.



The chart above displays the enthalpy of vaporization of a number of common solvents versus their boiling points. This provides a relative comparison of the overall volatility of the solvent and an indication of the relative ease of removal by distillation. Those solvents in the lower left hand corner require the least energy for removal. For a more complete listing of solvent enthalpies of vaporization, see the solvent properties table on page 6-6.

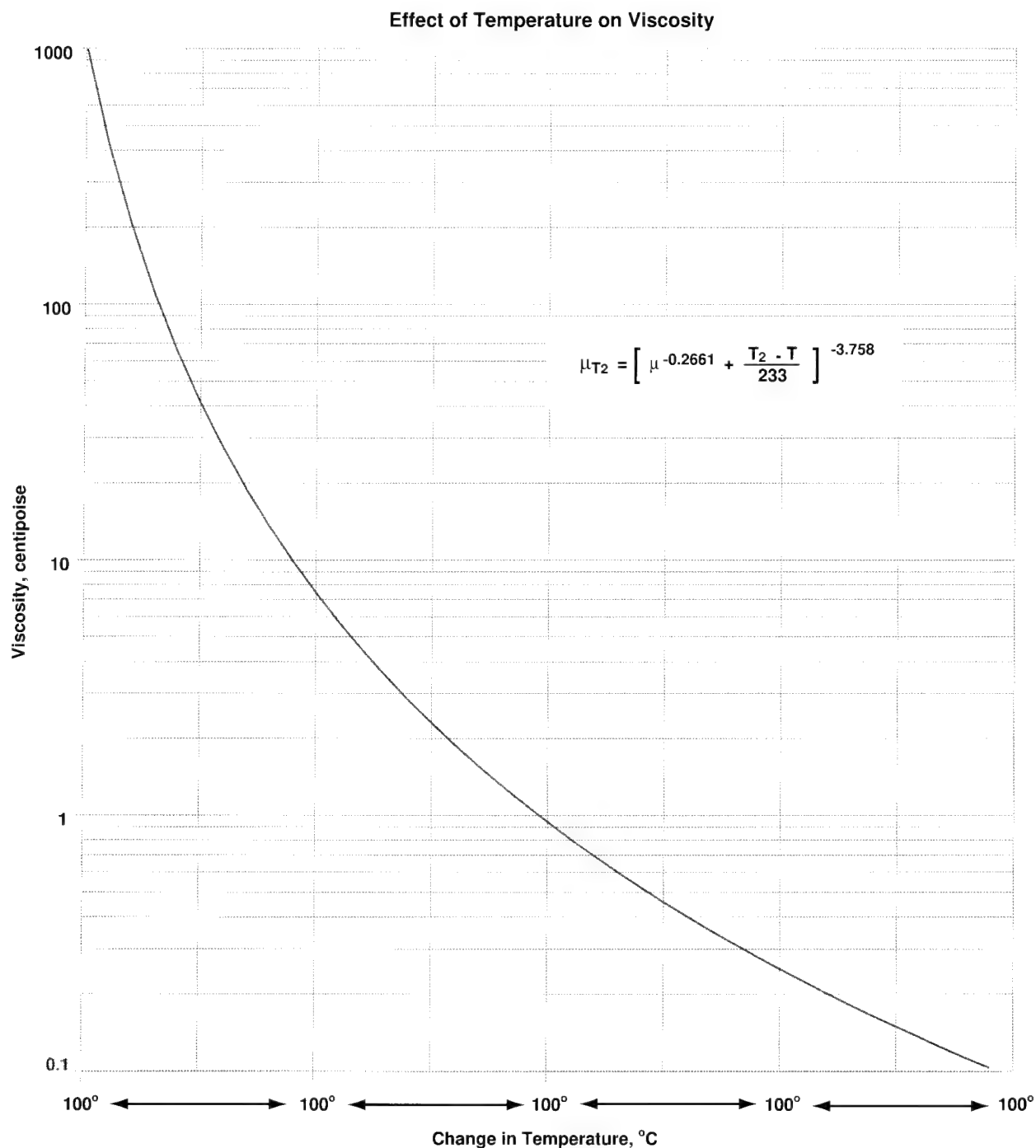
Effect of Temperature on Enthalpy of Vaporization



The enthalpy of vaporization (ΔH_{vap} , also called latent heat of vaporization) steadily decreases with increasing temperature and reaches zero at the critical temperature. ΔH_{vap} for several representative pure solvents is displayed above, as calculated using a modification of the Watson relation shown in the chart, from [206]. This allows calculation of enthalpy of vaporization at a second temperature ($\Delta H_{\text{vap},T_2}$) if the value is known at the normal boiling point (ΔH_{vap} at T_{BP}). ΔH_{vap} is usually reported in the literature at the normal boiling point. To use the equation, temperatures must be entered in °K (°K = °C + 273). When critical temperature (T_{C}) value is not known, the empirical Klincewicz equation, also from [206], may be used to estimate it based on the molecular weight (MW) and boiling point:

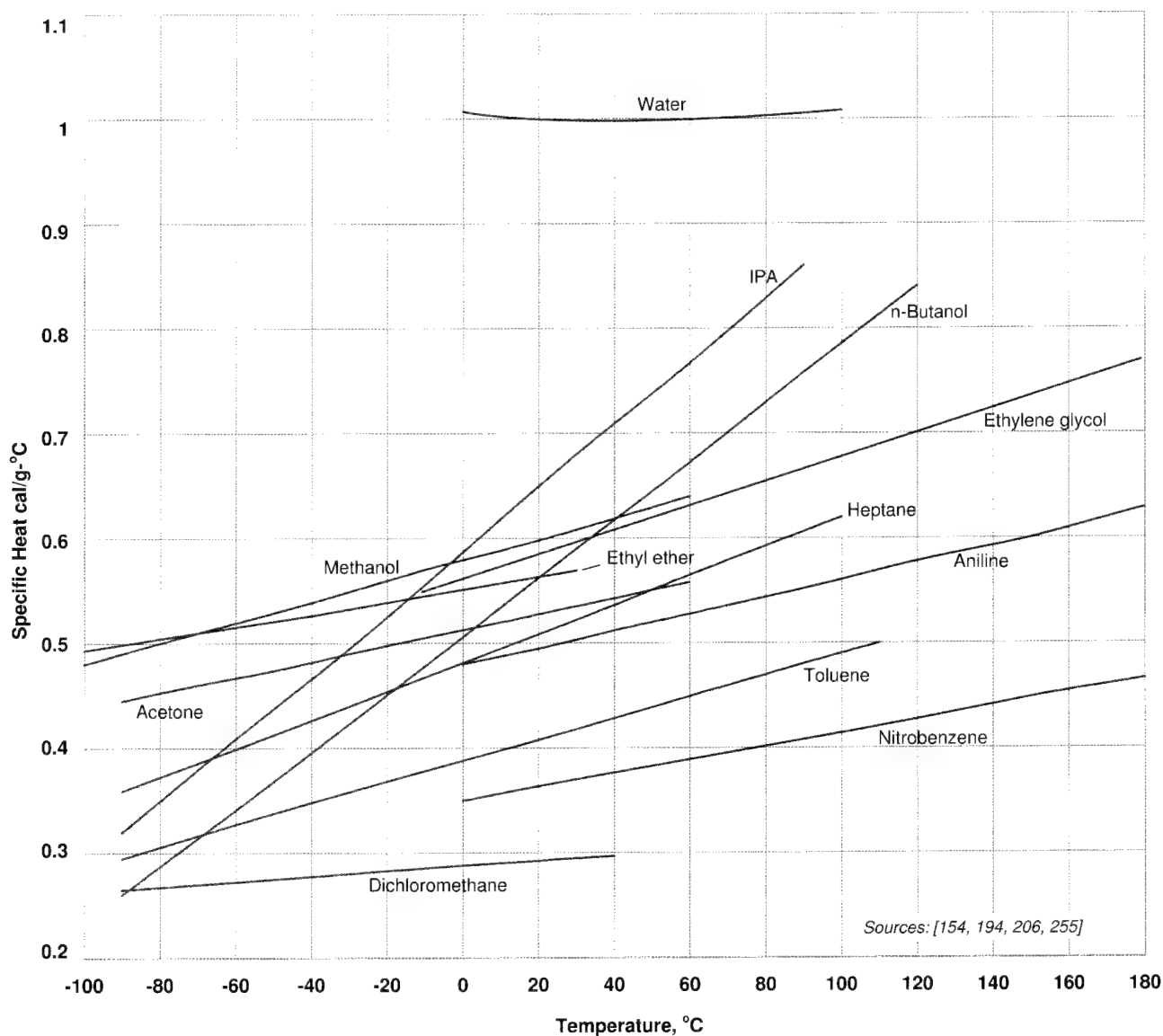
$$T_{\text{C}} = 50.2 - 0.16\text{MW} + 1.41T_{\text{BP}} \quad (\text{Temperature in K})$$

For very rough estimates of the increase in ΔH_{vap} over the normal liquid range of the solvent at atmospheric pressure, it can be assumed that for every 10°C drop in temperature, ΔH_{vap} increases by approximately 172 cal/gmole.



The viscosity of liquids increases as temperature decreases. The figure above shows a graph of viscosity vs. temperature change, based on the empirical relationship of Lewis and Squires as shown. The equation allows for a reasonably accurate estimate of viscosity of a liquid at a second temperature (μ_{T_2} at T_2) if the viscosity at a first temperature is known (μ at T). Viscosity is in cP, temperature is in °C. To use the chart, locate the known viscosity point on the curve, then shift over left or right by the appropriate temperature difference to obtain the new viscosity value. For more on the effects of viscosity on pressure drop through pipes and on pump performance, see page 3-25. To convert viscosity to kinematic viscosity, divide by the fluid density (see page 3-25 and 11-3). Chart adapted from [39, 206]

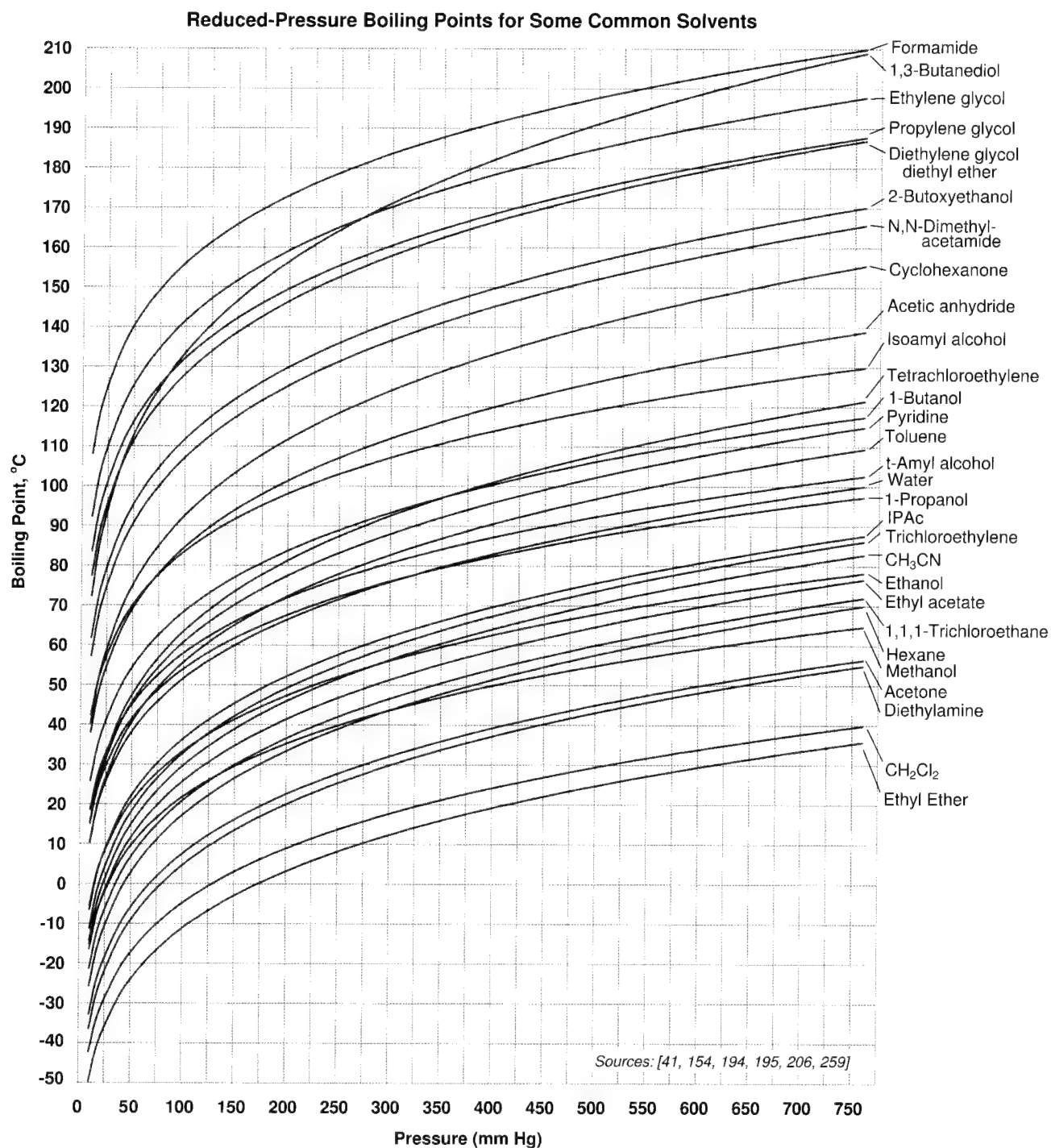
Effect of Temperature on Specific Heat



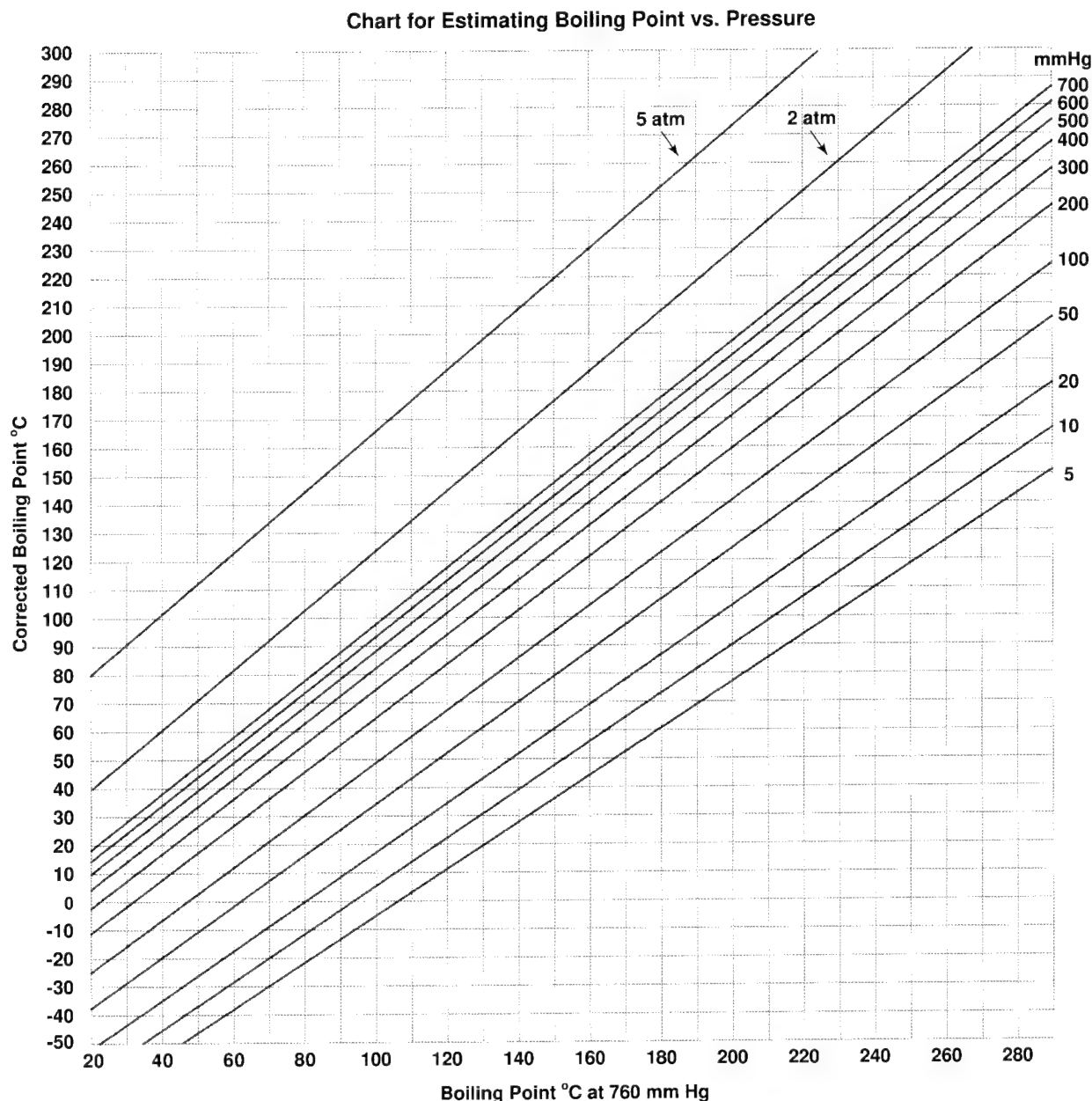
The chart above compares the specific heat (C_p , also called the isobaric heat capacity) of several representative solvents, on the basis of mass. As the chart shows, specific heat increases with temperature for most solvents.

There are no simple formulas to accurately predict the change in specific heat with temperature when the data are not available. Generally speaking, however, the specific heat of the “typical” organic solvent can be expected to increase by about 30% over its entire normal liquid range. The notable exceptions to this are water and most of the alcohols above methanol (ethanol, propanol, isopropanol, butanol, etc.), which exhibit a much higher change in specific heat over their normal liquid ranges (typically about 300%).

Note that while water has a relatively low molar specific heat, it has the highest specific heat per unit mass of most solvents. Therefore, designing heat transfer systems to accommodate a fluid with the thermal properties of water usually safely covers the capacity for other solvents. The specific heat of water does not change significantly between 0° and 100°C. For a closer look at the specific heat of water vs. temperature, see page 6-46.



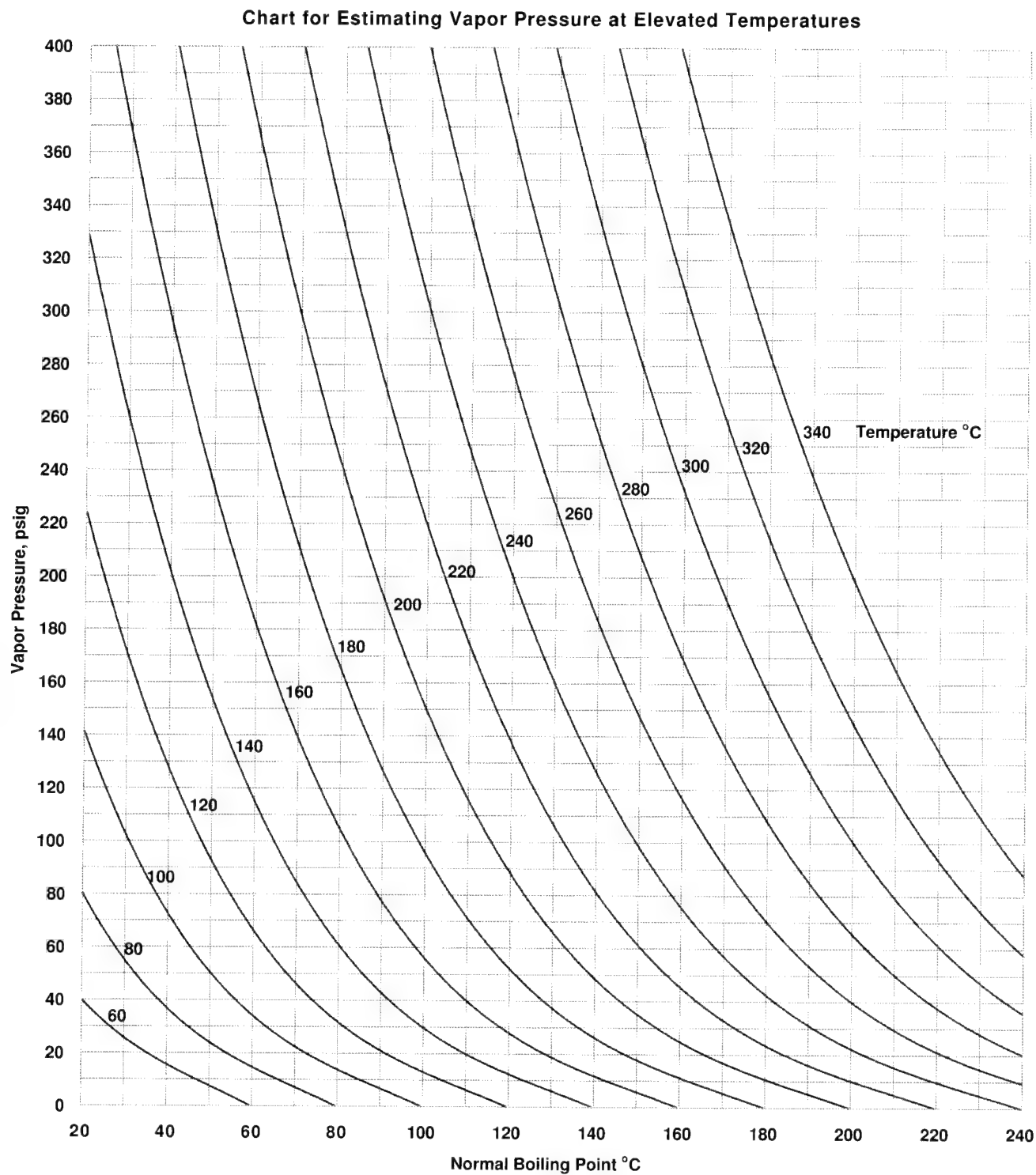
The chart above shows the approximate boiling points of some common solvents at pressures below 760 mm Hg (1 atm). To approximate the boiling point at nonstandard pressures for solvents not shown, see the chart on page 6-23.



Vapor-pressure temperature relationships have traditionally been expressed using the Antoine equation and lists of empirical constants. For most pure solvents, however, the figure above can be used to estimate the boiling point at the desired pressure if the boiling point at 760 mm Hg (1 atm) is known. The figure can also be used inversely to estimate atmospheric boiling point if the boiling point at another pressure is known. Extrapolations may also be made using the Cox equation:

$$\log P = A - \frac{B}{T + 230}$$

where P is in mmHg, T is in °C and A and B can be calculated from two known points on the pressure-temperature curve. The chart on the following page allows the estimation of vapor pressure at elevated temperatures. Chart derived from data in [41, 154, 194, 195, 259].



The chart above allows the user to approximate the increase in vapor pressure for most solvents at elevated temperatures. To use the chart, start on the x-axis at the normal boiling point (at 760 mmHg), project up to the desired temperature curve, then read left to find the estimated vapor pressure. Chart derived from data in [41, 154, 194, 195, 259].

Notes on Distillation and Azeotropes

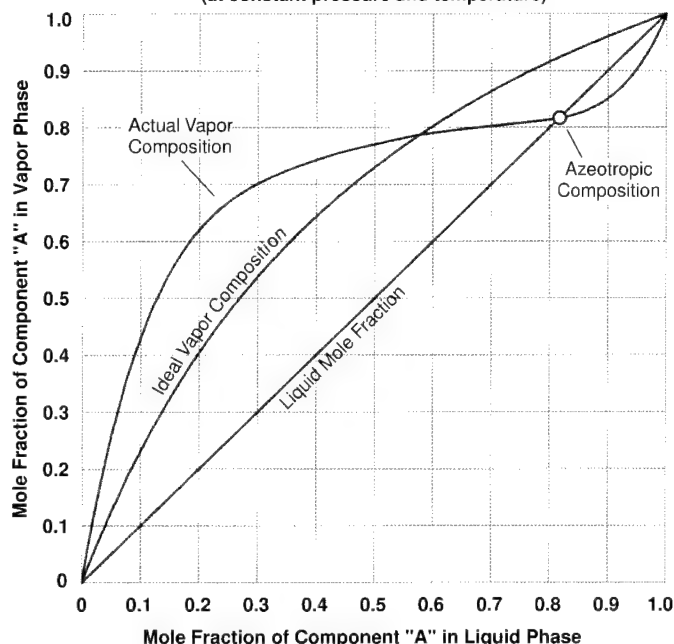
Distillation operations in fine chemical or pharmaceutical manufacturing are usually single-stage distillations designed to remove solvent for adjusting concentration, to remove water, or to exchange one solvent for another. This is called flash distillation, as opposed to distillation with reflux. Many common liquids form constant-boiling mixtures called **azeotropes** with other liquids, and it is often possible to take advantage of this fact to improve the efficiency of drying and solvent exchange operations by distillation.

Theory predicts that the composition of the vapor phase above a mixture of two liquids is determined only by the partial pressures and mole fractions of the two components (see the VLE at right). However, in reality, few mixtures follow such ideal behavior. Vapor composition can deviate significantly from the ideal because of the effects of polarity, hydrogen bonding, and other interactions. An azeotrope occurs if there is a point where the vapor curve crosses the $x=y$ diagonal, that is, *if the molar compositions of the liquid and vapor phases are equal*. Such mixtures always have a minimum or a maximum boiling point, meaning that the boiling point of the mixture at that composition is lower than or higher than the boiling points of either of the pure components. See the examples of the two-component boiling point diagrams below. Generally speaking, the more different two components are (i.e. an alcohol and a hydrocarbon), and the closer their boiling points, the more likely that they will form an azeotrope.

If the azeotrope occurs in a composition range where the two liquids are immiscible, the condensing vapor will form two separate liquid phases and the azeotrope is said to be heterogeneous. Heterogeneous azeotropes are always minimum boiling. In the case of immiscible solvents, the azeotropic boiling point can be predicted quite accurately using vapor pressure plots of the two components. The temperature at which the sum of the two individual vapor pressures equals the operating pressure of interest (say 760 mmHg) will be the azeotropic boiling point.

The tables on pages 6-28 to 6-43 list many common binary and ternary water-containing azeotropes that exist at atmospheric pressure for pure solvents. Note, however, that azeotropic composition is also a function of pressure as discussed on the following page. For more on vapor-liquid equilibria, distillation and azeotropes see [61, 113, 213, 234].

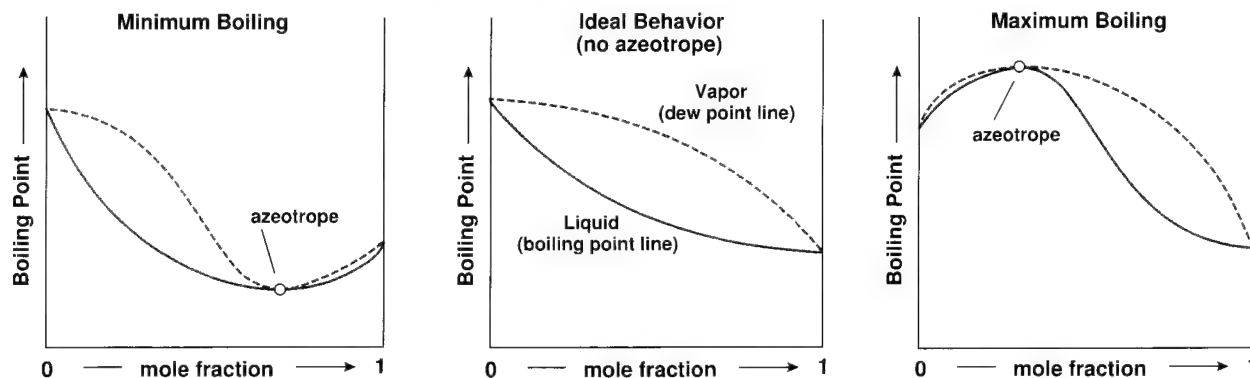
Typical Two-Component Vapor-Liquid Equilibrium (VLE) Diagram
(at constant pressure and temperature)

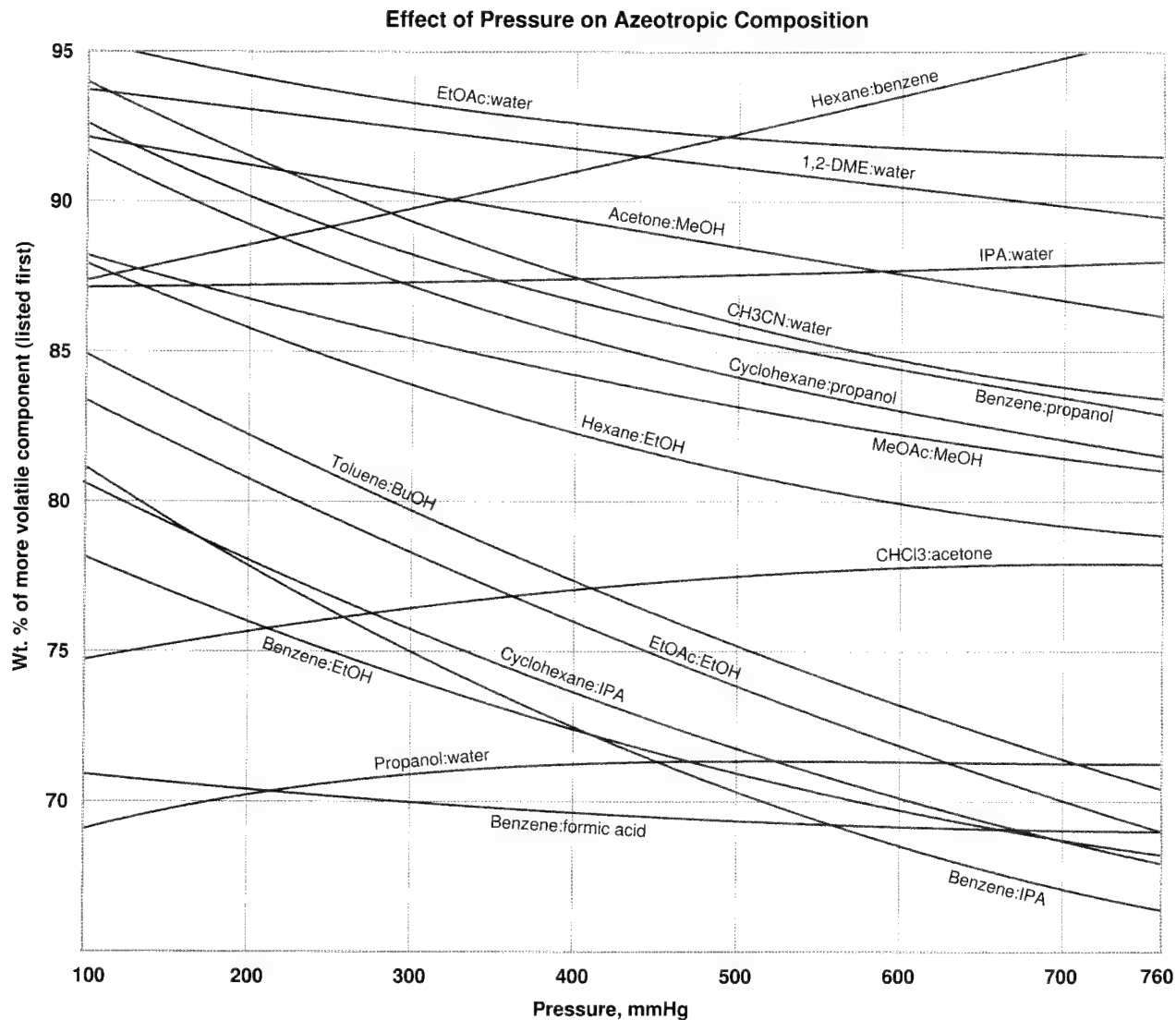


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The tables on pages 6-28 to 6-43 list many common binary and ternary water-containing azeotropes that exist at atmospheric pressure for pure solvents. Note, however, that azeotropic composition is also a function of pressure as discussed on the following page. For more on vapor-liquid equilibria, distillation and azeotropes see [61, 113, 213, 234].

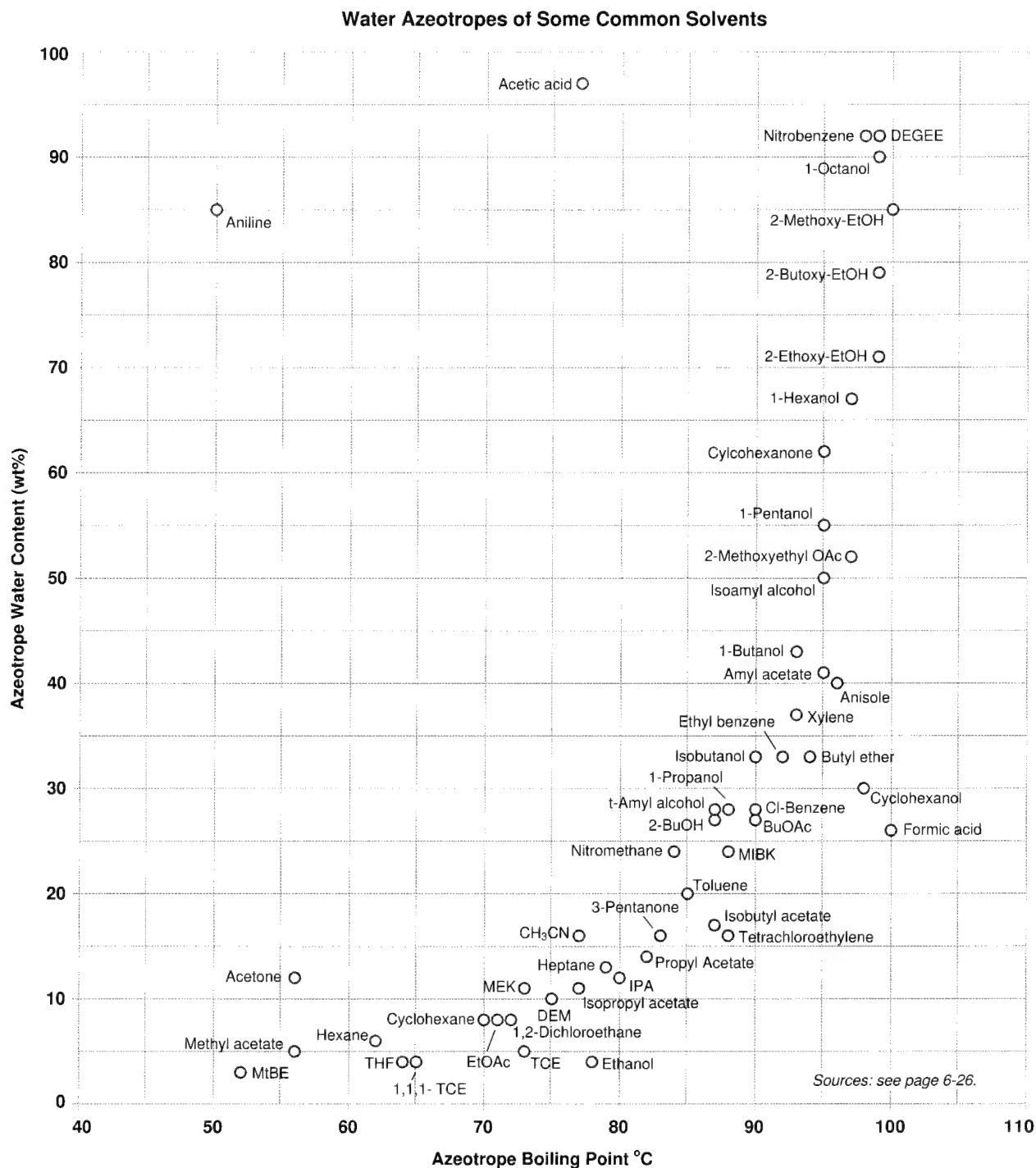
Example Boiling Point Diagrams for Two-Component Systems





The chart above indicates the change in azeotropic composition with pressure for some representative minimum-boiling binary mixtures. The compound pairs are listed in order of specific ΔH_{vap} (the compound with lower specific enthalpy of vaporization, in cal/g or Btu/lb, is listed first). In most cases, reducing the pressure enriches the vapor phase in the compound with the lower specific ΔH_{vap} , but there are many notable exceptions. Thus, when drying a solution by azeotropic removal of water, reducing the pressure will usually reduce the effectiveness of the operation as the vapor phase will contain less water. However, the formation of azeotropes involves many complex interactions at the molecular level, and thus vapor pressure, boiling point or ΔH_{vap} data are not enough to predict the azeotrope composition absolutely. On the other hand, azeotropic composition will usually not vary by more than 10% or so over the pressure range typically encountered in processing plants, i.e. a vacuum of about 100 mm Hg to atmospheric pressure. Chart sources: [108, 123, 234, 259].

Azeotrope Tables – The azeotropes tabulated on the following pages were taken from a variety of sources: [61, 105, 108, 109, 123, 145, 154, 234, 259]. In many cases there was considerable variation in the data. In other cases, extrapolations or interpolations were made to keep the data consistent. Thus, the values reported here should be considered approximate. Azeotropic composition will also be strongly affected by dissolved substances and the presence of other volatile components. Unless indicated otherwise, all values are for 760 mm Hg (1 atm).



In the above chart, azeotropic composition is plotted versus boiling point for the water azeotropes of a number of common solvents. For more information on binary and ternary water-containing azeotropes, see the tables which follow.

[illegible]

Pressure = 760 mm Hg (1 atm)

[illegible]

Binary Azeotropes of Some Common Solvents (continued)

B Component →																					
A Component ↓	Boil. Pt. °C	Wt% "A"																			
	65	33																			
	Ethylene Glycol	Formic Acid	Glycerol	n-Heptane	n-Hexane	1-Hexanol	Isoamyl Alcohol	Isobutanol	Isobutyl Acetate	Isopropanol	Isopropyl Acetate	Methanol	2-Methoxyethanol	2-Methoxyethyl Acetate	Methyl Acetate	Methyl Cyclohexane	Methyl Ethyl Ketone	Methyl Formate			
Acetic Acid		N		92 33	68 6		133 16				N	N			N						
Acetic Anhydride											N					99 18					
Acetone				56 90	50 59	N		N		N	N	56 88	N		56 48		N	N			
Acetonitrile				69 46	57 28			N		75 52	79 60	63 81	N		N	71 50					
Allyl Alcohol				85 37	65 4				N	N						85 42					
Amyl Acetate	148 94													145 8							
t-Amyl Alcohol				92 27	68 4								N	N		92 40					
Aniline	181 76			N	N	N	N									N					
Anisole	150 89					151 63	N					N	N	N							
Benzene	N	71 69		80 99	68 5	N	N	79 92		72 66	N	58 61	N		N	N	78 62				
1-Butanol	N			94 18	68 3			N	115 50	N	N	N	N	N		95 20	N				
2-Butanol				88 37	67 8			N	N	N		N	N			89 41	N				
t-Butanol				78 62	64 22			N				N				79 66					
2-Butoxyethanol	N				N	N						N					N				
Butyl Acetate	N			N			126 83	N		80 48		N	118 52	N	N						
Butyl Ether	140 94	N					130 35	N					122 32								
Butylamine					70 25					85 40		N									
Chlorobenzene	130 94	94 41		N	N	N	124 66	107 37		N		N	119 47	N	N	N	N				
Chloroform	N	59 85		N	60 72			N		61 96		53 87	N		65 77	N	80 17	N			
Cyclohexane	N	71 70		N	N		N	78 86		69 68	79 75	45 63	80 18		55 17	N	N				
Cyclohexanol	N			N	N	N										N					
Cyclohexanone						156 94							N								
Cyclopentane	46 84														43 62				26 54		
1,2-Dichloroethane	77 86			81 76	N		N	83 94		73 61	N	60 65				N	N				
Dichloromethane				N	N			N		N		38 93			N		N	N			
DietG Eth Ether Acetate																					
Diethoxymethane				88 96	N			N		80 48	88 58	63 35				N					
Diethylamine					N							70 39									

Binary Azeotropes of Some Common Solvents (continued)

A Component ↓	B Component →																	
	Acetic Acid	Acetic Anhydride	Acetone	Acetonitrile	Allyl Alcohol	Amyl Acetate	t-Amyl Alcohol	Aniline	Anisole	Benzene	1-Butanol	2-Butanol	t-Butanol	2-Butoxyethanol	Butyl Acetate	Butyl Ether	Butylamine	Chlorobenzene
Diethylene Glycol										N								
Dieth.Glyc Ethyl Ether								N										
Dieth.Glyc Methyl Ether								N										
Dimethoxymethane			N							N								
Dimethyl Sulfide	N		N										N					
Dimethyl Sulfoxide			N	N						N	N							
N,N-DMF	159 74		N	N						N	N			N	N			N
1,4-Dioxane	119 20		N	N	N		101 80			82 12	N	99 60	N					N
Ethanol	N		N	73 56						68 32	N	N	N	N	N	N	82 49	N
2-Ethoxyethanol						N			135 94	N	N			N	126 36	127 50		127 32
Ethyl Acetate	N		N	75 77	N					77 94	N	N	76 75	N	N			N
Ethylbenzene	115 34			N			N	N		N	115 33	N	28 5	N	N	N		N
Ethyl Ether	N		N	N						N	N							N
Ethylene Glycol						148 6		181 24	150 11	N	N			N	N	140 6		130 6
Formic Acid	N		N	N						71 31						N		94 59
Glycerol																		
n-Heptane	92 67		56 10	69 54	85 63		92 73	N		80 1	94 82	88 63	78 38		N			N
n-Hexane	68 94		50 41	57 72	65 96		68 96	N		68 95	68 97	67 92	64 78	N			70 75	N
1-Hexanol			N					N	151 37	N				N				N
Isoamyl Alcohol	55 81							N	N	N					126 17	130 65		124 34
Isobutanol			N	N						79 8	N	N	N		N	N		107 63
Isobutyl Acetate											115 50	N						
Isopropanol			N	75 48	N					72 34	N	N	N		80 52		85 60	N
Isopropyl Acetate	N	N	N	79 40						N	N							
Methanol	N		56 12	63 19					N	58 39	N	N	N	N	N		N	N
2-Methoxyethanol			N	N			N		N	N	N	N			118 48	122 68		119 43
2-Methoxyethyl Acetate						145 92	N		N		N				N	138 30		N
Methyl Acetate	N		56 52	N						N					N			N

N = non-azeotrope

Pressure = 760 mm Hg (1 atm)

Chloroform	N	N	N	N	59	78	N	N	59	60	N	61	53	65
Cyclohexane	N	80	25	65	31	72	N	N	71	N	78	69	45	55
Cyclohexanol	N	N	N	N	N	N	N	N	30	N	14	32	37	83
Cyclohexanone	N	N	N	N	N	N	N	N	N	N	N	N	N	N
Cyclopentane	40	62	37	88	45	71	74	N	46	81	83	73	60	43
1,2-Dichloroethane	45	59	N	40	2	43	N	41	77	N	6	39	35	N
Dichloromethane	N	N	N	40	2	N	N	30	14	N	N	N	7	N
Dieth Glyc Ether Acetate	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Diethoxymethane	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Diethylamine	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Diethylene Glycol	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Dieth Glyc Ether	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Dieth Glyc Methyl Ether	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Dimethoxymethane	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Dimethyl Sulfide	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Dimethyl Sulfoxide	N	N	N	74	42	N	N	N	88	N	80	52	63	N
N,N-DMF	N	N	N	74	42	N	N	N	88	N	80	52	63	N
1,4-Dioxane	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Ethanol	N	N	N	74	42	N	N	N	88	N	80	52	63	N
2-Ethoxyethanol	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Ethyl Acetate	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Ethylbenzene	N	N	N	74	42	N	N	N	88	N	80	52	63	N
Ethyl Ether	N	N	N	74	42	N	N	N	88	N	80	52	63	N

Binary Azeotropes of Some Common Solvents (continued)

B Component →																				
Boil. Pt. °C	65																			
Wt% "A"	33																			
A Component ↓	Ethylene Glycol	Formic Acid	Glycerol	n-Heptane	n-Hexane	1-Hexanol	Isoamyl Alcohol	Isobutanol	Isobutyl Acetate	Isopropanol	Isopropyl Acetate	Methanol	2-Methoxyethanol	2-Methoxyethyl Acetate	Methyl Acetate	Methyl Cyclohexane	Methyl Ethyl Ketone	Methyl Formate		
Diethylene Glycol			N																	
Dieth.Glyc Ethyl Ether	192 55																			
Dieth.Glyc Methyl Ether	192 70																			
Dimethoxymethane		N			N					N		42 92			N			N		
Dimethyl Sulfide		N								N		34 88					N	29 38		
Dimethyl Sulfoxide	N				N			N				N								
N,N-DMF	N	153 99		97 5	N					N		N	N		N		N	N		
1,4-Dioxane	N	113 57		92 44	60 2			101 96	N	N	N	78 9	N			94 45				
Ethanol	N			72 48	59 21		N	N		N	77 47	N	N		57 3		75 34	N		
2-Ethoxyethanol				97 14	N		N	N	N			N		N		99 15				
Ethyl Acetate				77 94	65 38			N		76 75		62 51	N		N	N	77 88			
Ethylbenzene	133 86	94 32		N	N	N	126 51	107 20	N	N		N	117 46	136 85			N			
Ethyl Ether		N			N	N				N		N			N			28 44		
Ethylene Glycol	-		N	98 3	N	N				N		N				N				
Formic Acid		-		78 57	61 28											80 47				
Glycerol	N		-																	
n-Heptane	98 97	78 43		-	N	N	98 93	91 73	N	76 50	88 33	59 46	92 77		57 4		77 30			
n-Hexane	N	61 72		N	-	N	N	68 98		63 77	69 81	50 74	N		52 39	N	64 70	N		
1-Hexanol	N			N	N	-								N		N				
Isoamyl Alcohol				98 7	N		-	N	N				N	N		98 13				
Isobutanol				91 27	68 2			-	107 55	N	N	N	N	N	N	93 32	N			
Isobutyl Acetate				N			N		-				116 84	N						
Isopropanol	N			76 50	63 23			N		-	80 52	N			N		78 32			
Isopropyl Acetate				88 67	69 9			N		80 48	-	65 20				89 78				
Methanol	N			59 54	50 26			N		N	65 80	-	N		54 18		64 70	N		
2-Methoxyethanol				92 23	N		N	N	116 16			N	-	N			N			
2-Methoxyethyl Acetate						N	N	N	N				N	-						
Methyl Acetate				57 96	52 61			N		N		54 82			-		N			

Pressure = 760 mm Hg (1 atm)

[illegible]

Binary Azeotropes of Some Common Solvents (continued)

B Component →																					
Boil. Pt. °C	65																				
Wt% "A"	33																				
A Component ↓	Acetic Acid	Acetic Anhydride	Acetone	Acetonitrile	Allyl Alcohol	Amyl Acetate	t-Amyl Alcohol	Aniline	Anisole	Benzene	1-Butanol	2-Butanol	t-Butanol	2-Butoxyethanol	Butyl Acetate	Butyl Ether	Butylamine	Chlorobenzene			
Methyl Cyclohexane	99 82			71 49	85 58		92 60	N		N	96 80	90 59	79 34					N			
Methyl Ethyl Ketone			N							78 38	N	N		N	N			N			
Methyl Formate			N																		
Methyl Iodide			42 95																		
Methyl Isobutyl Ketone			N							N	114 70			N							
N-Methyl Pyrrolidone			N															N			
Methyl t-Butyl Ether													N								
Nitrobenzene	N							N		N	N	N		N				N			
Nitromethane	101 96		N	N	89 43		93 50			79 13	98 71	91 46	79 32					N			
1-Octanol								184 17													
n-Pentane	N		33 80	35 90			N			N	N	N	36 97					107 63			
1-Pentanol																					
3-Pentanone					96 28		101 60			N	N	98 42	N								
P G Meth Ether Acetate																					
1-Propanol	N		N	81 28	97 74					77 17	N	N			94 40			97 83			
Propyl Acetate	N		N	N	95 52		100 58			N	N	97 48									
Propylene Glycol								180 43		N					N						
Propylene Oxide					N																
Pyridine	138 49	N	N	N	N		N			N	119 30	89 68			N			N			
Tetrachloroethylene	107 61		N	N	93 55		101 27			N	110 68	97 43	N	N	121 79	120 65		N			
Tetrahydrofuran			64 8	N						N	N	N						N			
Toluene	101 72		N	81 20	92 50		100 44	N		N	106 72	95 45	N	N	N			N			
1,1,1-Trichloroethane	86 96									N								N			
Trichloroethylene	87 96		N	75 71	81 84		87 93			N	87 97	84 85	76 67		N			N			
Triethylamine	163 31		N	71 63						N											
Water	77 97		56 12	77 16	88 27	95 41	87 28	50 85	96 40	69 9	93 43	87 27	80 12	99 79	90 27	94 33	N	90 28			
Xylenes	116 27		N	N	N		N	N	N	N	115 27	N	N	N	N	N		N			

Pressure = 760 mm Hg (1 atm)

[illegible]

Binary Azeotropes of Some Common Solvents (concluded)

B Component →																				
Boil. Pt. °C	65																			
Wt% "A"	33																			
A Component ↓	Ethylene Glycol	Formic Acid	Glycerol	n-Heptane	n-Hexane	1-Hexanol	Isoamyl Alcohol	Isobutanol	Isobutyl Acetate	Isopropanol	Isopropyl Acetate	Methanol	2-Methoxyethanol	2-Methoxyethyl Acetate	Methyl Acetate	Methyl Cyclohexane	Methyl Ethyl Ketone	Methyl Formate		
Methyl Cyclohexane	N	80 53		N	N	N	98 87	93 68			89 22									
Methyl Ethyl Ketone				77 70	64 30			N		78 68		64 30	N		N		-			
Methyl Formate					N							N					-			
Methyl Iodide		42 94			N					42 98		38 96			N			31 17		
Methyl Isobutyl Ketone				98 13				108 9		N		N	114 75				N			
N-Methyl Pyrrolidone												N								
Methyl t-Butyl Ether												52 85								
Nitrobenzene	186 41		N		N	N						N		N						
Nitromethane	N	97 54		80 35	62 21	N	101 88	94 58	N	79 28	N	64 8				81 40				
1-Octanol	184 63					N														
n-Pentane	N	34 90		N	N		N	N		35 94		31 93			34 88	N	N	22 47		
1-Pentanol																				
3-Pentanone		105 67		93 35	N		N	102 80		N		N				95 40				
P G Meth Ether Acetate																				
1-Propanol	N			85 35	66 4		N	N	N	N		N	N		N	86 35	N			
Propyl Acetate					N			101 83				N								
Propylene Glycol																				
Propylene Oxide																				
Pyridine	N	150 36		96 25	N		N	N				N			N					
Tetrachloroethylene	119 94	88 50			N		116 81	103 60	116 47	82 19		64 36	109 76				N			
Tetrahydrofuran	N			N	63 54			N		N		61 69								
Toluene	110 98	86 50	N	N	N	N	110 90	101 55	N	81 31	N	64 29	106 75	N	N	N	N			
1,1,1-Trichloroethane					67 29							56 78								
Trichloroethylene	N	74 75		N	N		N	85 91		76 70		69 62				N	N			
Triethylamine				N	N							N				N				
Water	N	100 26		79 13	62 6	97 67	95 50	90 33	87 17	80 12	77 11	N	100 85	97 52	56 5		73 11	N		
Xylenes	135 93	94 28	N	N	N	140 85	127 48	108 12		N		N	120 45	140 60	N		N			

Pressure = 760 mm Hg (1 atm)

Methyl Iodide	31 83	-	N	30 60	N	94 35	115 60 114 48	111 97	88 24
	N								
Methyl Isobutyl Ketone	N	-	N			94 35	115 60 114 48	111 97	88 24
N-Methyl Pyrrolidone						N			N
Methyl t-Butyl Ether									52 3
Nitrobenzene	N		N	N					98 92
Nitromethane	81 60	N	N	35 99	99 45	90 52 98 55	101 15 95 20	97 45	84 24 N
1-Octanol									99 90 N
n-Pentane	N	22 53 30 40	35 1		N	27 57	N	N	35 1 N
1-Pentanol									95 55
3-Pentanone	95 60		99 55		95 57 101 60	N		N	83 16
P G Meth Ether Acetate									
1-Propanol	86 65	N	90 48		95 43	94 60	N	94 52 N	88 28 97 7
Propyl Acetate			98 45		101 40	94 40		N	82 14
Propylene Glycol								111 98	N
Propylene Oxide				28 43					N
Pyridine		115 40	100 85		N	N	113 51	110 78	94 42 N
Tetrachloroethylene	N	114 52	95 80	N		94 48	113 49	N	88 16 N
Tetrahydrofuran				N		N	N	N	64 4
Toluene	N	111 3	97 55	N	N	93 49 N	110 22 N	-	85 20 N
1,1,1-Trichloroethane						73 7		N	65 4
Trichloroethylene	N		81 20		N	82 17	N		73 5
Triethylamine	N								76 10
Water	73 89	N	88 76	52 97 98 8	84 76	99 10	94 58	88 84	-
Xylenes	N			95 45	83 84	88 72 82 86	94 58 88 84 64 96	85 80	93 37

Ternary Water-Containing Azeotropes of Some Common Solvents

B Component →																									
Boil. Pt. °C	75																								
Wt% "A"	43																								
Wt% H ₂ O	6																								
A Component ↓	Acetic Acid	Acetone	Acetonitrile	Allyl Alcohol	t-Amyl Alcohol	Benzene	1-Butanol	2-Butanol	t-Butanol	Butyl Acetate	Butyl Ether	Butylamine	Chlorobenzene	Chloroform	Cyclohexane	1,2-Dichloroethane	Dichloromethane	Diethoxymethane	Diethylamine	Dimethoxymethane	Ethanol	2-Ethoxyethanol	Ethyl Acetate	Ethyl Ether	
Acetic Acid	-																								
Acetone		-	N											60 38 4							N				
Acetonitrile		N	-			66 23 8				N									N		73 44 1				
Allyl Alcohol				-		68 9 9									66 81 8										
t-Amyl Alcohol					-	N									N										
Benzene		66 69 8	68 82 9	N		-		75 88 7	67 71 8												65 74 7				
1-Butanol						69 2 10	-	N		91 8 29	91 35 30	N									N				
2-Butanol						75 5 7	N	-	N		87 56 25														
t-Butanol						67 21 8		N	-						65 21 8										
Butyl Acetate			N			91 63 29				-															
Butyl Ether						91 35 30		87 19 25			-														
Butylamine							N					-									82 50 8				
Chlorobenzene													-								77 14 7				
Chloroform	N	60 58 4	N																		78 91 4				
Cyclohexane				66 11 8	N				65 71 8												62 76 7				
1,2-Dichloroethane																					68 77 7				
Dichloromethane																					N				
Diethoxymethane																					73 69 13				
Ethanol		N	73 55 1			65 19 7	N					82 42 8	77 80 7	78 5 4	63 20 5	68 16 7	N	73 18 13			-	N	70 8 9	N	
Ethyl Acetate																					70 83 9		-		

Pressure = 760 mm Hg (1 atm)

	N	N	Formic Acid
			Glycerol
69 33 6		78 8 41 76 22 11	n-Heptane
56 12 3		60 5 5 62 3 19	n-Hexane
		N	1-Hexanol
N		N	Isoamyl Alcohol
	N	N	Isobutanol
			Isobutyl Acetate
	75 19 10	83 47 13	Isopropanol
		N	Isopropyl Acetate
N		N	Methanol
			2-Methoxyethanol
			Z Methyl Acetate
		77 22 12	Methyl Cyclohexane
73 14 11	N	N	Methyl Ethyl Ketone
			Methyl Isobutyl Ketone
		N	Methyl t-Butyl Ether
			Nitromethane
		N	n-Pentane
			1-Pentanol
			3-Pentanone
			PG Meth Ethr Acetate
	N	69 82 9	1-Propanol
			Propyl Acetate
		N	Pyridine
			Tetrachloroethylene
74 37 12		81 31 15	Z Toluene
67 16 6		71 13 8	Trichloroethylene
75 15 10		67 31 6	Triethylamine
			Xylenes

Ternary Water-Containing Azeotropes of Some Common Solvents (concluded)

B Component →		<div>Boil. Pt. °C</div> <div>Wt% "A"</div> <div>Wt% H₂O</div>			A Component ↓																									
		<div>75</div> <div>43</div> <div>6</div>																												

Solvents Limited For Pharmaceutical Use

CLASS 1 - Solvents to be avoided (known or suspected carcinogens)		
Benzene	1,2-Dichloroethane	Carbon tetrachloride
1,1-Dichloroethane	1,1,1-Trichloroethane	

CLASS 2 - Solvents to be limited (neurotoxins or teratogens)		
Acetonitrile	1,4-Dioxane	Nitromethane
Chlorobenzene	2-Ethoxyethanol	Pyridine
Chloroform	Ethylene glycol	Sulfolane
Cyclohexane	Formamide	Tetralin
1,2-Dichloroethene	Hexane	Toluene
Dichloromethane	Methanol	1,1,2-Trichloroethene
1,2-Dimethoxyethane	2-Methoxyethanol	Xylene
N,N-Dimethylacetamide	Methylbutyl ketone	
N,N-Dimethylformamide	Methylcyclohexane	

CLASS 3 - Solvents considered to have low toxic potential at normal pharmaceutical levels		
Acetic Acid	Ethyl Ether	Methyl-t-butyl ether
Acetone	Ethyl Formate	Methylethyl ketone
Anisole	Formic Acid	Methylisobutyl ketone
1-Butanol	Heptane	Pentane
2-Butanol	Isobutanol	1-Pentanol
Cumene	Isobutyl acetate	1-Propanol
Dimethylsulfoxide	Isopropyl acetate	2-Propanol
Ethanol	Methyl acetate	Tetrahydrofuran
Ethyl acetate	3-Methyl-1-butanol	

The U.S. Food and Drug Administration requires that residual levels of the above solvents in pharmaceuticals be closely monitored in terms of allowable daily exposure or intake. For more details and specific limits, see [90, 94].

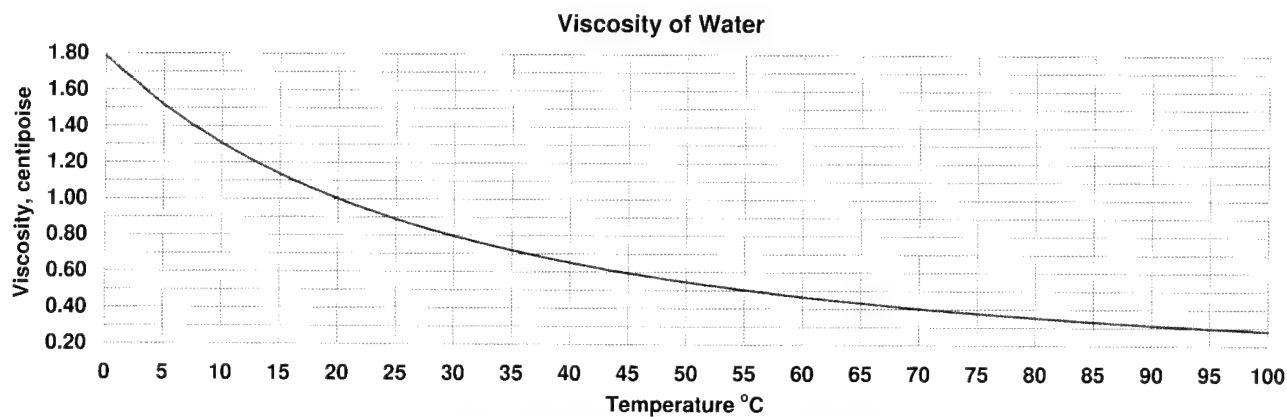
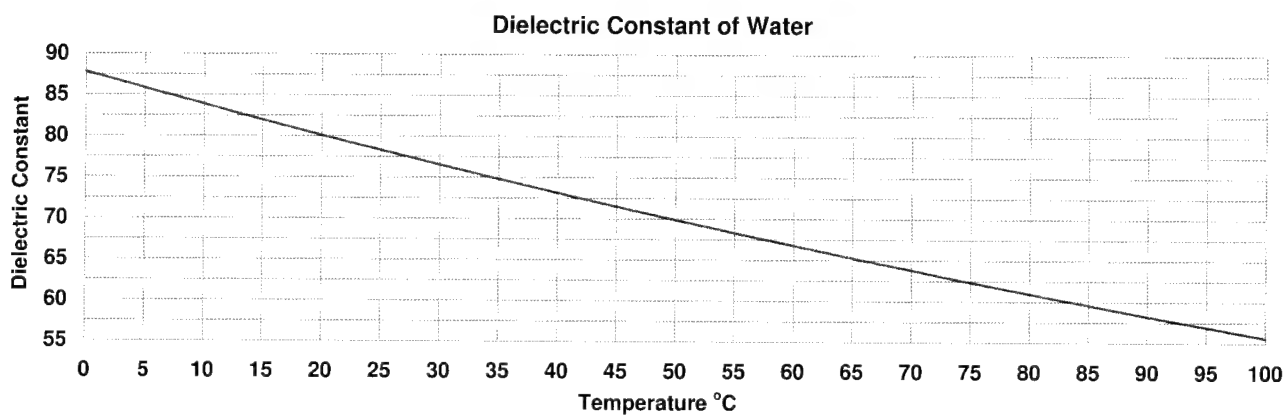
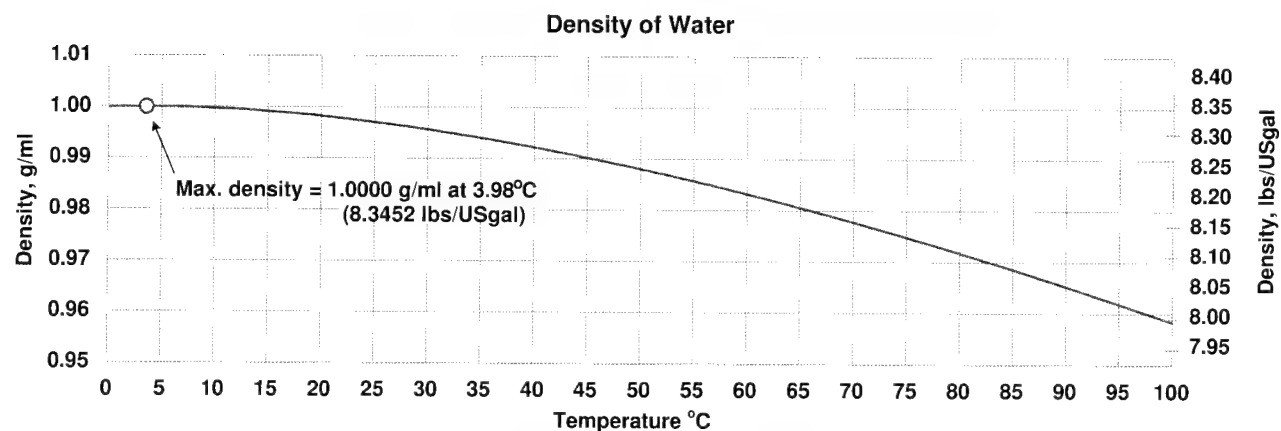
Common Types of Denatured Ethanol

Grade	Denaturant Charge (volumes added to 100 volumes of pure EtOH)		Denaturant volume %
SDA-1	Methanol	4	3.81
	MIBK	1	0.95
SDA-3A	Methanol	5	4.76
SDA-3C	Isopropanol	5	4.76
SDA-12A	Toluene	5	4.76
SDA-13A	Ethyl ether	10	9.09
SDA-19	Ethyl ether	100	50
SDA-20	Chloroform	5	4.76
SDA-23A	Acetone	7	7.41
SDA-30	Methanol	10	9.09
SDA-32	Ethyl ether	5	4.76
SDA-35A	Ethyl acetate	4.25	4.08
SDA-39C	Ethyl acetate	1	0.99
SDA-44	n-Butanol	10	9.09
ACS Reagent Alcohol*	Methanol	5.27	4.75
	Isopropanol	5	5

*prepared by adding 5 volumes IPA to 100 volumes SDA-3A.

Denatured alcohol is pure ethanol that has been rendered unfit for drinking by the addition of substances such as methanol, ethyl acetate, IPA and others in quantities specified by the federal government. Over 50 types of **specialty denatured alcohol** (SDA) are used in industry, with denaturants ranging from gasoline to clove oil. Many SDAs are derivatives of two main types: SDA-1 and SDA-3. Most are available as 200 proof (meaning pure solvent, not diluted by water) and in diluted forms (for example, 190 proof: 95% of the SDA, 5% water). See [197] for more information.

Important Temperature Dependent Properties of Water

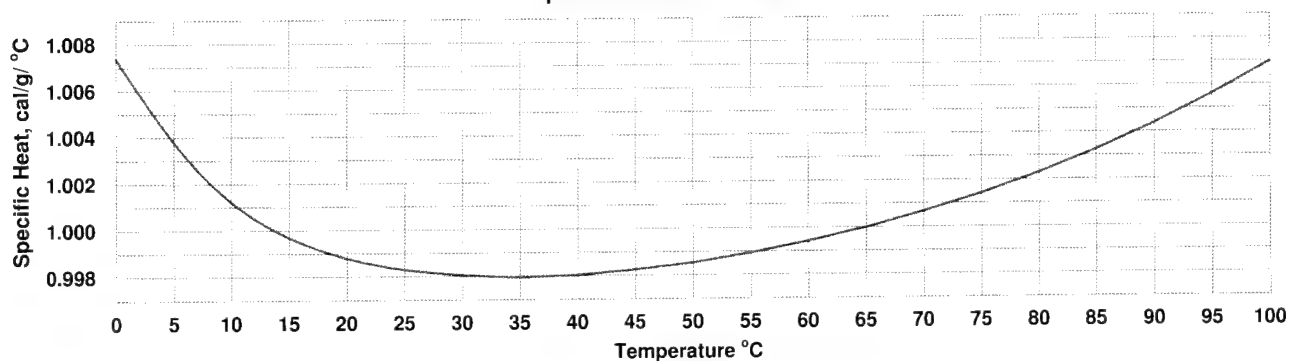


Other Selected Properties of Water (20°C)

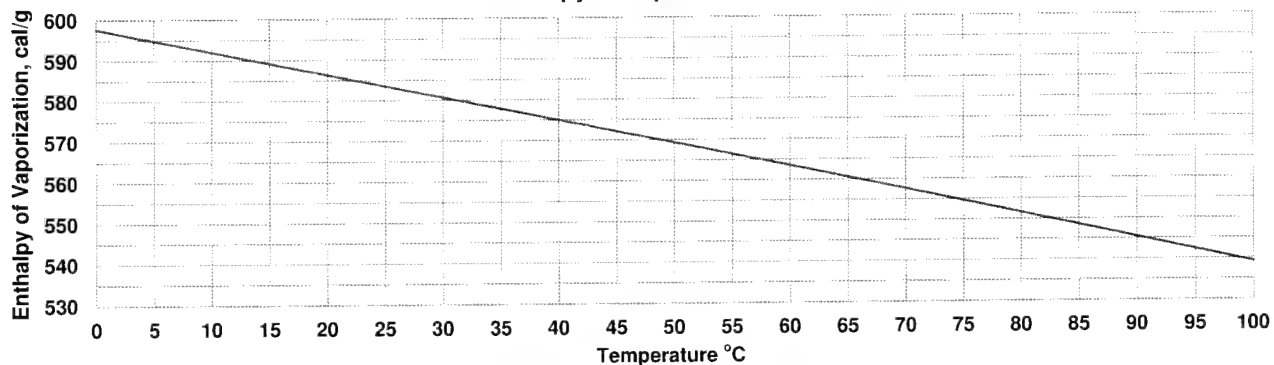
Vapor Pressure	17.5 mm Hg	Pumping Head	0.43 psi/ft or 2.3 ft/psi
Critical Temperature	374.2 °C		
Critical Pressure	218.4 atm		
Critical Density	0.323 g/cm ³	Density	1.00 g/cm ³
Surface Tension	72.8 dynes/cm		998.2 kg/m ³
Dipole Moment	1.85 debye		8.33 lbs/USGal
			3.78 kg/USGal
			6.23 lbs/ft ³

Sources: [54, 194, 195, 250]

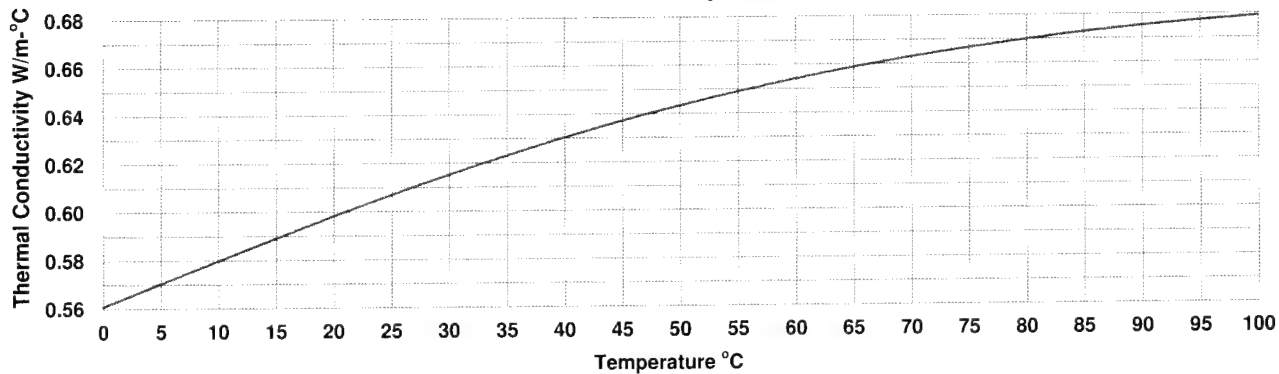
Specific Heat of Water



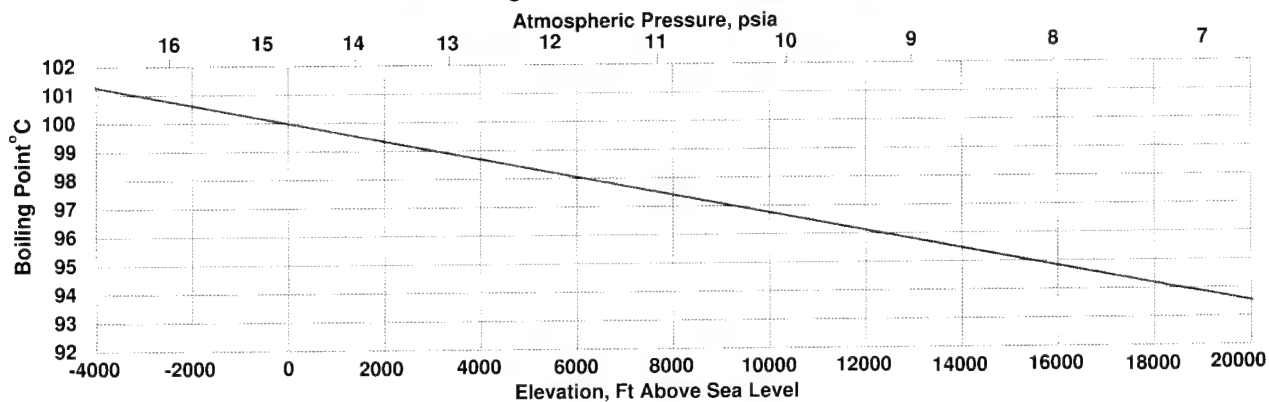
Enthalpy of Vaporization of Water



Thermal Conductivity of Water



Boiling Point of Water vs. Elevation



Common Water Purification Methods

Treatment Method ↓	Acidity	Alkalinity	Biofilm	Carbon Dioxide	Calcium, Magnesium	Chlorine, Chloramines	Inorganics	Dissolved Iron	Microbiologicals	Organics	Particulate Matter	Pyrogens	Silica
Activated Carbon Filter						x			x				
Chemical Addition	x	x	x	x	x	x					x		
Clarifier (sediment and turbidity filter)											x		
Dealkalizer		x											
Degasifier				x									
Deionization		x		x	x		x	x					x
Distillation		x					x	x	x	x	x	x	
Birm or Magnesium Greensand								x					
Sanitization			x			x							
Organic Scavengers										x			
Reverse Osmosis (RO)		x			x		x	x	x	x	x	x	x
Softening Filter (ion exchange)					x			x					
Neutralizing Filter	x												
Submicron Filtration									x		x		
Ultraviolet Light						x			x	x			
Ultrafiltration									x	x	x	x	
Multimedia Filtration											x		

Source: [250]

The table above lists some of the most common water purification methods. Some of the properties requiring treatment include: **Acidity** – often due to the spontaneous formation of carbonic acid from CO_2 absorbed from the air. **Alkalinity** – due to the presence of OH^- , CO_3^{2-} , and HCO_3^- negative ions. **Hardness** – caused by the presence of di- and tri-valent metallic ions such as Ca, Mg, Fe and Mn and HCO_3^- ions. “Softening” can be accomplished by ion exchange resins or by treatment with lime or soda ash, which precipitate many metal ions.

Process Water Quality Specifications

The quality of water required for any operation depends on the specifics of the process. Two commonly used sets of water quality specifications are shown below.

ASTM Reagent Grade

Type	I	II	III	IV
Conductivity 25°C (micromhos/cm)	0.056	1.0	0.25	5.0
Resistivity 25°C (megohms/cm)	18	1.0	4.0	0.2
Total Silica ($\mu\text{g/L}$)	3	3	500	-
Total Organic Carbon ($\mu\text{g/L}$)	100	50	200	-
Chlorides ($\mu\text{g/L}$)	1	5	10	50
Sodium ($\mu\text{g/L}$)	1	5	10	50
pH	-	-	-	5.0 - 8.0

Type	A	B	C
Max. Heterotrophic Bacteria Count (cfu)	10/1000ml	10/100ml	10/10ml
Endotoxin, EU/ml	<0.03	0.25	N/A

USP 24 Pharmaceutical Grade

Total Organic Carbon	<0.5 ppm	Type	Purified Water	WFI
Conductivity at 25°C	1.3 micromhos/cm (or >0.81 megohms/cm resistivity)	Bacteria (guideline only)	100 cfu/ml	10 cfu/100ml
		Endotoxin, EU/ml	-	<0.25

EU = International Endotoxin Units cfu = colony forming units WFI = water for injection

Source: [250]

Water Conductivity and Resistivity

Resistivity megohms/cm (MΩ/cm)	Conductivity micromhos/cm (μS/cm)	ppm as NaCl at 25°C	ppm as CaCO ₃ at 25°C
0.00026	3860	2000	1700
0.00034	2930	1500	1275
0.0005	1990	1000	850
0.00099	1020	500	425
0.0024	415	200	170
0.0032	315	150	127.5
0.0048	210	100	85
0.0095	105	50	42.05
0.023	42.7	20	17
0.031	32.1	15	12.7
0.05	20	8	10
0.1	10	4	5
0.2	5	2	2.5
0.3	3.28	1.5	1.27
0.45	2.21	1	0.85
0.88	1.13	0.5	0.42
1	1	0.4	0.5
2	0.5	0.2	0.25
2.65	0.38	0.15	0.13
3.7	0.27	0.1	0.085
6.15	0.16	0.05	0.042
10	0.1	0.04	0.05
11.5	0.087	0.015	0.012
13.1	0.076	0.01	0.008
15.2	0.066	0.005	0.004
16.9	0.059	0.002	0.002
17.6	0.057	0.001	0.001
18.24	0.055	0	0

Sources: [205, 213, 250]

Conductivity (and its inverse, resistivity) is one of the most common measurements of water purity. A resistivity of approximately 18 MΩ/cm is considered the standard for pure, ion-free water. The table above lists values for conductivity, their corresponding resistivities, and their NaCl and CaCO₃ equivalents.

CaCO₃ equivalents – Water purity data is usually not reported in units of molarity or normality, but rather in a common measurement of solution strength called the “CaCO₃ equivalent”. Substances are reported in mg/L (equal to ppm) as CaCO₃, even if the substance being measured bears no relation to CaCO₃ whatsoever (the rationale for this was that the molecular weight of CaCO₃ ~100). The actual concentrations of substances can be converted to amounts as CaCO₃ by the use of conversion factors based on stoichiometric principles (see the explanation below). Thus equal CaCO₃ amounts of different substances represent equal stoichiometric reaction quantities. For example 50 mg/L Na as CaCO₃ will react with 50 mg/L Cl as CaCO₃ to form 50 mg/L NaCl as CaCO₃.

The factors for conversion to CaCO₃ can be calculated by dividing the molecular weight of CaCO₃ (100.09) by that of the species of interest. For example, the conversion factor for anhydrous CuSO₄ (molecular weight = 159.60) is:

$$100.09 / 159.60 = 0.63$$

Thus, 100 mg/L CuSO₄ = (100) (0.63) = 63 mg/L CuSO₄ as CaCO₃.

7 Compressed Gases

Contents

SAFETY

Notes on the Safe Handling of Compressed Gases	7-2
Special Precautions for Gaseous Hydrogen	7-3
Precautions for Cryogenic Liquids	7-3

CYLINDER AND CONNECTION DATA

Estimating Partial-Cylinder Contents	7-3
Common Gas Cylinder Types and Specifications	7-4
Typical Gas Cylinder Markings	7-4
Cylinder Valve Outlet Connections for Common Gases	7-6
Recommended Torque Values for CGA Outlet Connections	7-7
CGA Fitting Specifications	7-8

GAS METERING

Metering Gases	7-9
Heating Gas Cylinders Safely	7-9
Care and Use of Gas Pressure Regulators	7-10

GAS PROPERTIES

Physical Properties of Gases	7-11
Properties of Some Cryogenic Liquids	7-12
Gas Leak Detection	7-13

AIR PROPERTIES

Properties of Air	7-14
Dew Point vs. Relative Humidity	7-15

COMPRESSED AIR

Compressed Air Systems	7-16
Compressor Horsepower Requirements	7-17
Typical Compressed Air Usage Rates	7-17
Compressed Air Tank Capacity	7-18
Equivalent Cubic Feet of Compressed Air	7-18
Air Flow Through Pipes and Tubes	7-19
Pressure Drop Through Fittings for Air, Steam, or Gases	7-19
Air Flow Through Orifices	7-20

VACUUM

Vacuum Systems	7-21
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Notes on the Safe Handling of Compressed Gases

All compressed gases are potentially hazardous because of the high pressures at which they are delivered. A sudden release of such pressure can cause injuries by propelling a cylinder across a room or whipping a line. Other hazards include possible asphyxiation from using inert gases in a confined area, fire, explosion, chemical burns or poisoning depending on the gas. Before using any compressed or liquefied gas, always read the appropriate Material Safety Data Sheet (MSDS) to understand the properties of the product, the specific health, fire and explosion hazards, and the precautions to be taken for its safe handling, storage and use. Always identify the contents of a cylinder by the label before using it – never rely on cylinder color alone for identification.

Handling and Storage – Protect cylinders and valves from mechanical shock. Never drop, drag or slide cylinders. Store cylinders in a cool, dry well ventilated area. Always secure cylinders with straps or chains. Never store cylinders near radiators, heat sources or live electrical circuits. If outside, protect cylinders from weather extremes and damp ground to prevent rusting. Separate full cylinders from empty cylinders and separate oxygen and other oxidizers from flammable gases by at least 20 feet or a fire-resistant wall. Rotate stock.

Temperature – Never expose any part of a compressed gas cylinder to temperatures greater than 125°F (52°C) or less than -22°F (-30°C). Never expose a cylinder to flames or sparks. Never heat a cylinder to raise the pressure without using an approved cylinder warming system (see page 7-9). Placing cylinders in warm water baths for this purpose is not recommended because of the possibility of corrosion and eventual rupture.

Cylinder Use – Never remove the cap from a cylinder until the cylinder is fully secured. Caps protect the valve from damage and, in the event of valve failure, prevent the cylinder from toppling over by venting the escaping gas out both sides. Never lift a cylinder by the cap. Never attempt to force a stuck cap or valve – contact the supplier.

Always use regulators and pressure relief devices when connecting cylinders to systems with lower pressure ratings. Always confirm that the CGA fittings are appropriate for the labeled product (see page 7-6). Use only regulators approved for the specific gas and CGA outlet connection. Check the outlet connection for debris or mechanical damage before connecting the cylinder to the regulator or other components. See page 7-10 for more on the proper use of regulators. Always open the cylinder valve slowly. Leak test all systems prior to use. Cylinder valves should be closed and the pressure relieved from the system whenever you anticipate an extended idle period. Exercise extreme caution when relieving pressure of corrosive gases such as HCl to the atmosphere.

Lines and equipment should be made of materials compatible with the gases being used. Use check valves or traps to prevent backflow of water or other contaminants into the cylinder. If backflow occurs, mark the cylinder “contaminated” and notify the supplier immediately.

Never return product to or refill a compressed gas cylinder. Cylinders may be refilled only by qualified producers. Never introduce another material into a cylinder. Never attempt to mix gases in a cylinder. Never take a cylinder to zero pressure – always leave about 25 psig of residual pressure. Never remove product identification labels or change cylinder color. Use approved perforated labels to indicate cylinders that are full, in-use, or empty.

Never use oxygen as a substitute for compressed air. Use only oxygen-compatible threading compounds such as Teflon tape on systems for oxygen or oxidizer service. When using flammable gases, always properly ground and bond the cylinders and connecting lines. When using corrosive gases, always wear the appropriate personal protective equipment, and be sure to purge the regulator and system with inert gas immediately after use.

Returning Cylinders – When returning an empty cylinder, close the valve before shipment, and replace the valve cap and any valve outlet caps or plugs originally shipped with the cylinder. Label the cylinder “Empty”. In the event of valve or cylinder damage, mark it clearly before returning the cylinder to the supplier.

More information on the use of compressed and liquefied gases can be found at the product stewardship section of the Air Products and Chemicals Co. website www.airproducts.com. As always, contact your gas supplier with any questions about special situations or unfamiliar circumstances.

Special Precautions for Gaseous Hydrogen

Hydrogen is a particularly dangerous gas. It is colorless, odorless, highly flammable and burns with an almost invisible flame. It can form explosive mixtures with air, O₂, Cl₂ and many other gases. Because of its low minimum ignition energy, it may ignite in the absence of any apparent ignition source. Even the static built up by an escaping leak can be sufficient to cause ignition. Its small molecular size makes it prone to leaking, even from systems that may be tight with respect to other gases. Piping systems must be designed in accordance with state and local requirements, based on the standards set out in NFPA Pamphlet 50-A [185]. All hydrogen cylinders must be properly grounded and bonded. Hydrogen has especially wide flammability limits in air (4-75%). As with any flammable gas, leaking cylinders should be moved outdoors and the vendor contacted immediately. If hydrogen is used on a regular basis, an electronic hydrogen monitoring system is strongly recommended.

Precautions for Cryogenic Liquids

General – Liquefied cryogenic gases, typically nitrogen (LIN), argon (LAR), helium (LHE), and oxygen (LOX) are supplied in special double walled insulated cylinders designed for relatively low pressures. These cylinders must always be stored and transported in an upright position only. If a cylinder falls over, carefully stand it back up and contact the supplier before attempting to use it.

Special Hazards – Human tissue easily freezes at the temperatures at which cryogenic liquids are delivered (typically below -90°C). Always wear insulated gloves when handling cylinder valves or other components. Always wear a full face shield and gloves when transferring liquid product.

Because of the constant automatic venting of gas (up to 3% of the contents/day) as the cylinder absorbs heat from the environment, liquefied inert gas cylinders must be stored in well ventilated areas to prevent the buildup of dangerous oxygen-deficient atmospheres. In areas of regular use, such as NMR laboratories where liquid helium is used, oxygen sensors should be installed to warn of low oxygen concentrations. Never transport cryogenic gas cylinders in occupied elevators. Likewise, storing Liquid Oxygen (LOX) cylinders in a confined space can result in an oxygen-enriched atmosphere, which greatly increases the potential for a fire. Dispense such products only in well ventilated areas.

Cylinder Use – Cryogenic liquids have liquid to gas expansion ratios on the order of 1:800. To prevent dangerous pressure buildup, handling systems must be designed so that no liquefied product becomes trapped in a non-vented space. Be aware that cryogenic liquid temperatures are sometimes low enough to cause air to liquefy and freeze in lines or vent tubes, creating a possible blockage.

Cylinders are fitted with a gas withdrawal valve or a liquid withdrawal valve or both, as well as other specialized automatic venting equipment. Always be familiar with the design and function of the components of the system you are using before attempting to withdraw product. Never attempt to change the outlet connection valves fitted by the supplier. Doing so can create an extremely dangerous situation.

Estimating Partial-Cylinder Contents

For most gases, there is a roughly linear relationship between cylinder contents and pressure. Thus, if the starting amount (weight) of product and the starting pressure are known (this information should be available from the supplier) then the remaining contents of a partially used cylinder may be estimated using the following formula:

$$\% \text{ of product remaining} = \frac{\text{Current pressure} \times \text{Full cylinder Capacity}}{\text{Full cylinder pressure}}$$

This relationship does not hold true, however, for liquified gases. Liquid product vaporizes to maintain equilibrium at its vapor pressure in the cylinder as gas is consumed, and thus the pressure in the cylinder remains relatively constant until the cylinder is practically empty. A reliable estimate of the remaining contents can be made only by weight. The table of gas properties on page 7-11 indicates which gases are normally supplied as liquified products.

Common Gas Cylinder Types and Specifications

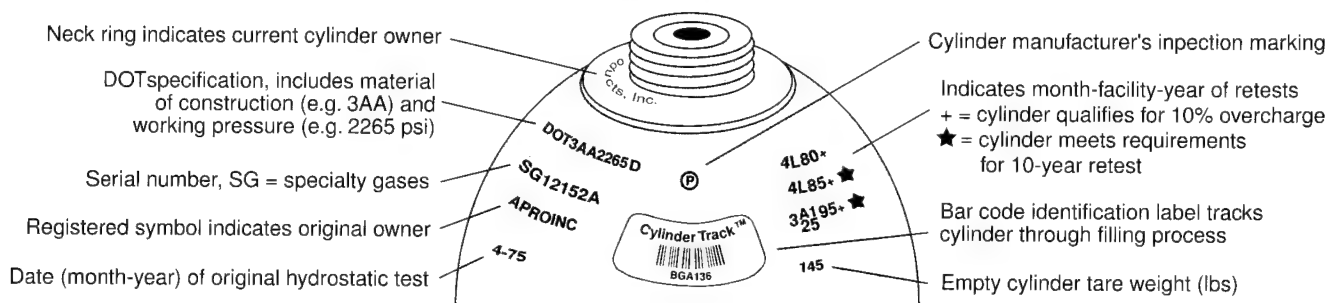
Compressed gas cylinders come in many shapes and sizes and are supplied by many major manufacturers. The chart below lists some of the specifications for a number of the common types used in industry. The chart includes cylinder dimensions, U. S. Department of Transportation shipping specification, and designations given by various manufacturers. The primary listing is the designation given by the Messer Gas Technology and Services Group [176]. The diagrams on the opposite page provide information on cylinder tare weights and the internal volume as well as a visual indication of relative cylinder sizes. The actual capacity of each cylinder type depends on the properties of the gas it contains.

Cylinder Specification and Conversion Chart

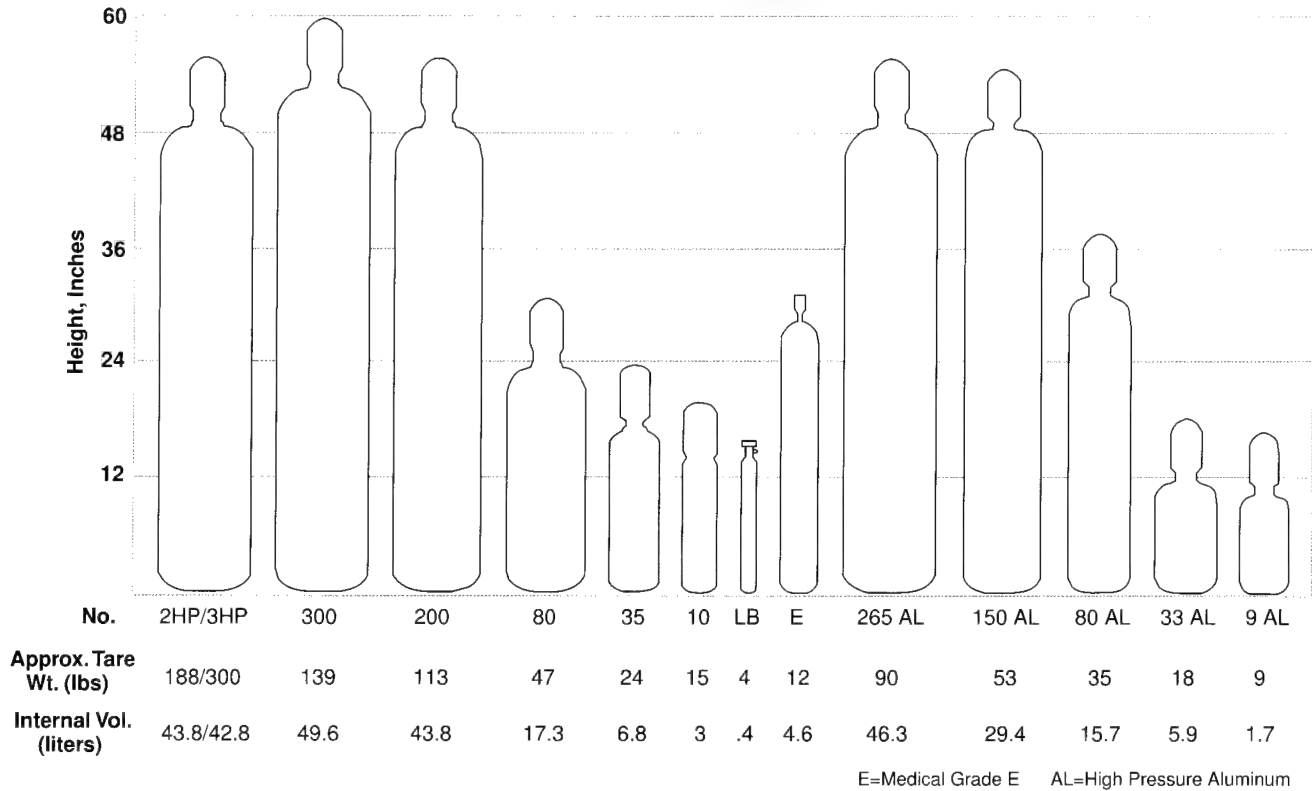
Messer GT&S Cylinder No.	Nominal Diam. x Height (inches)	DOT Specification	Corresponding Cylinder Designation for Other Major Suppliers							
			Air Products	Airgas	Air Liquide (Alphagaz)	Matheson Gas	Scott Specialty Gases	Praxair	BOC Gases (Airco)	AGA Gases
2HP	9 x 56	3AA3500	BY	2HP	44H	1H	-	3K	-	-
3HP	10 x 56	3AA6000	BX	3HP	44HH	1U	-	6K	500	485
300	9 x 60	3AA2400	A	300	49	1L	K	T	300	049
200	9 x 56	3AA2015/ 3AA2265	B	200	44	1A	A	K	200	044
80	7 x 37	3AA2015	C	80	16	2	B	Q	80	016
35	7 x 23	3AA2015	D-1	35	7	3	C	G	30	007
10	4 x 21	3AA2015	D	7	3	4	D	F	12	003
LB	2 x 15	3E1800	LB	LB	LB	LB	LB	LB/RB	LB	LB
LX	2 x 16	3E1800	LG	LX	LBX	7	-	ELB	7X	-
E	4.5 x 31	3AA2015	E	E	MEDE	3L	ER	ANE	E	005
265 AL	10 x 57	3AL2216	A(AL)	-	47(AL)	-	-	ALT	265A	-
150 AL	8 x 53	3AL2215	B(AL)	150A	30(AL)	1R	AL	ALS	152A	A31
80 AL	7 x 38	3AL2216	C(AL)	80A	22(AL)	2R	BL	ALQ	82A	A16
33 AL	7 x 21	3AL2216	D-1(AL)	33A	7(AL)	3R	CL	ALH	32A	A07
9 AL	4.5 x 15	3AL2215	D(AL)	9A	-	-	-	-	9A	-
350	15 x 48	4BA240	A1	350	110	1F	XL	FX	410	110
400	15 x 56	4AA480	AA	400	125	1K	XG	FA	-	126
65	10 x 53	4BA300	A3	65	55	1J	XP	FC	55	-
380	12 x 48	8AL	A(C ₂ H ₂)	380	70	1B	XF	380	5	161
140	7 x 38	8AL	-	-	-	-	-	-	-	-
50	8 x 45	4BA240	A3	-	-	-	-	-	-	-
25	12.5 x 18.5	4BA240	LP5	25	-	-	-	-	-	-
DM3	9 x 16.3	39NRC	-	D227	-	-	SCOTTY III	-	212L	-
DM1	3 X 11	4BA240	X100	-	4D	-	SCOTTY II	-	-	-
DAL	4 X 16.25	39NRC	-	-	8AL	-	SCOTTY IV	-	-	-
DM2	3 X 10.75	39NRC	-	-	7EOC	-	MINI-MIX	-	-	-

Sources: [6, 170, 176]

Typical Cylinder Markings



High-Pressure Cylinders



Low-Pressure Cylinders

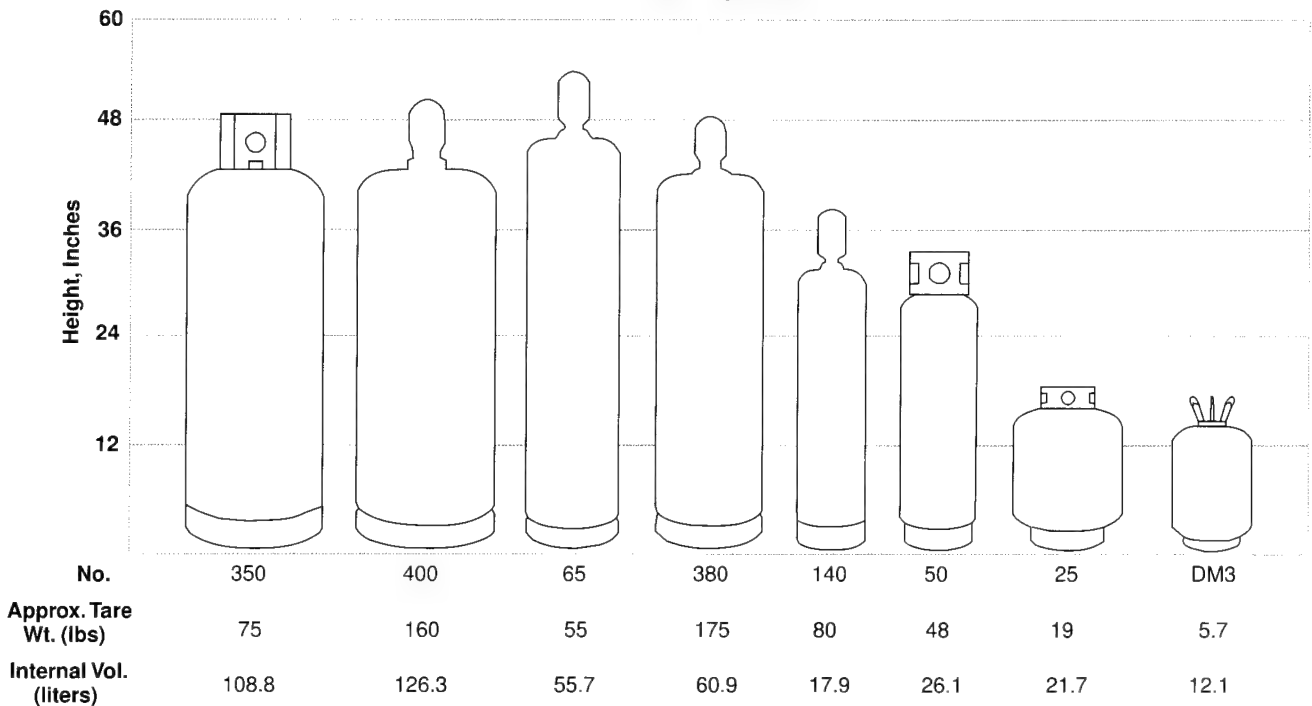


Diagram Courtesy MG Industries

Cylinder Valve Outlet Connections for Common Gases

Compressed gas cylinder valve outlet connections are specified by the Compressed Gas Association (CGA), Standard V-1 (for more information see www.cganet.com). Threads and seat styles vary widely and only regulators that match the CGA outlet connection code number can be used on a given cylinder. Fitting types for some common gases are listed below. Recommended torque values for some CGA fittings are provided on the following page. More detailed dimensions and specifications for some common CGA connection types are provided on the page 7-8.

Cylinder Valve CGA Outlet Connections for Common Gases

Acetylene	510	Ethylene	350	Methyl Chloride	510
Air (industrial)	590	Ethylene Oxide	510	Methyl Mercaptan	330
Air (breathing air)	346	Fluorine	679	Monoethylamine	705
Allene	510	Dichlorodifluoromethane (Freon 12)	660	Monomethylamine	705
Ammonia	705/240	Chlorotrifluoromethane (Freon 13)	660	Natural Gas	350
Argon	580	Bromotrifluoromethane (Freon 13B1)	660	Neon	580
Arsine	350	Tetrafluoromethane (Freon 14)	580	Nickel Carbonyl	660
Boron Trichloride	660	Chlorodifluoromethane (Freon 22)	660	Nitric Oxide	660
Boron Trifluoride	330	1,2-Dichlorotetrafluoroethane (Freon 114)	660	Nitrogen	580
Bromine Pentafluoride	670	Hexafluoroethane (Freon 116)	660	Nitrogen Dioxide	660
Bromine Trifluoride	670	Octafluorocyclobutane (Freon RC318)	660	Nitrogen Trioxide	660
Bromotrifluoroethylene	510	Dichlorofluoromethane	660	Nitrosyl Chloride	330
1,3-Butadiene	510	Fluoroform	660	Nitrous Oxide	326
Butane	510	Monochloropentafluoroethane	660	Oxygen	540
Butenes	510	1,1-Difluoroethane	510	Perfluor-2-butene	660
Carbon Dioxide	320	1,1-Difluoroethylene	350	Perfluoropropane	660
Carbon Monoxide	350	Germane	350	Phosgene	660
Carbonyl Fluoride	750	Helium	580	Phosphine	350
Carbonyl Sulfide	330	Hexafluoroacetone	330	Phosphorous Pentafluoride	330
Chlorine	660	Hexafluoropropylene	660	Propane	510
Chlorine Trifluoride	670	Hydrogen	350	Propylene	510
Chlorotrifluoroethylene	510	Hydrogen Bromide	330	Silane	350
Cyanogen	750	Hydrogen Chloride	330	Silicon Tetrafluoride	330
Cyanogen Chloride	750	Hydrogen Fluoride	670	Sulfur Dioxide	660
Cyclopropane	510	Hydrogen Selenide	350	Sulfur Hexafluoride	590
Deuterium	350	Hydrogen Sulfide	330	Sulfur Tetrafluoride	330
Diborane	350	Iodine Pentafluoride	670	Sulfuryl Fluoride	660
1,2-Dibromodifluoromethane	668	Isobutane	510	Tetrafluoroethylene	350
Dimethylamine	705	Isobutylene	510	Trimethylamine	705
Dimethylether	510	Krypton	580	Vinyl Bromide	510
2,2-Dimethyl Propane	510	Methane	350	Vinyl Chloride	510
Ethane	350	Methyl Acetylene	510	Vinyl Fluoride	350
Ethyl Acetylene	510	Methyl Bromide	330	Vinyl Methyl Ether	510
Ethyl Chloride	510	3-Methyl-1-butene	510	Xenon	580

Courtesy of Victor High Purity Gases

Recommended Torque Values for Sealing CGA Outlet Connections

CGA Connection Number	Recommended Torque ft-lbs	Maximum Torque ft-lbs	CGA Connection Number	Recommended Torque ft-lbs	Maximum Torque ft-lbs
110 (washer)	10	15	450	40	55
165	8	10	500	35	50
170 (washer)	10	15	510	35	50
180 (washer)	10	15	520	35	50
182	15	25	540	40	60
200	25	35	555	40	60
280	25	35	580	40	60
290	30	45	590	40	60
295	25	35	621	35	50
296	35	50	622	35	50
300	35	50	624	35	50
320 (washer)	20	30	625	35	50
326	25	35	626	35	50
330 (washer)	20	30	660 (washer)	30	45
346	35	50	670 (washer)	30	45
350	35	50	678 (washer)	25	35
410	35	50	679 (washer)	25	35
440	40	55	705 (washer)	40	60

Recommended Torque Values for Bullet Nose Connections

Valve Material	Nipple Material	Recommended Torque ft-lbs
Brass	Brass	35 - 45
Brass	Stainless Steel	35 - 50
Stainless Steel	Brass	35 - 50
Stainless Steel	Stainless Steel	35 - 60
"Hand-Tight" - all materials		8 - 15

Torque Conversion Factor: ft-lbs x 1.35 = NM (newton-meters)

Recommended Torque Values for Gasket Connections

Gasket Material	Recommended Torque ft-lbs
Fiber	20 - 30
PTFE	15 - 25
CTFE	20 - 35
Lead	30 - 45
Copper	35 - 45

Data provided by Air Products, Inc. [6]

The tables above provide general torque guidelines for CGA fittings based on tests with new components. Do not rely on these figures alone to ensure a good seal. Always leak-test systems after assembly. If you find that significantly higher torques are necessary, inspect parts for damage or wear, and replace or repair the faulty components. Applying excessive force can damage sealing surfaces and gall threads, and force gasket materials, especially PTFE, into the opening and block it.

Never use pipe dope on CGA connections. In the case of bullet nose connections, the seal takes place not at the threads, but at the circle of contact between the valve seat and nipple. This is designed to be a dry metal seal. For gasket connections, since gaskets flatten upon tightening, use a new gasket each time the system is assembled. Gasket connections are more susceptible to leaking than bullet nose connections if components shift or vibrate. Therefore be sure to support or secure the system or protect it from mechanical stress. "Hand-tight" connections seal by means of a soft nipple or soft O-ring on the nipple. Tightening with tools is not necessary and may damage the seal. Teflon tape may be used on the threads of any of these connections to protect the threads and prevent galling during assembly or disassembly.

CGA Fitting Specifications

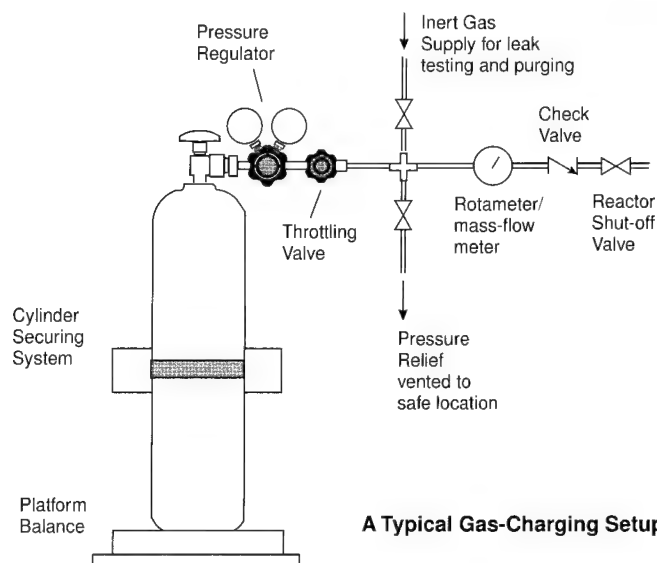
The table below lists specifications for a number of common CGA connection types, including thread count and size, thread direction and fitting type. More detailed information on CGA connections can be found in the Compressed Gas Association technical bulletin TB-14, available from the CGA or at www.cganet.com.

Dimensions and Specifications for Some Common CGA Fitting Types

CGA No.	Style	Outside Thread Diam (in.)	Thread count and size	Left or Right handed	Internal or External Threads
160	1/4" SAE Flare	1/8	27 NGT	RH	INT
165		0.4375	20 UNF-2A	RH	EXT
170		0.5825	18 UNF-2A	RH	EXT
180		0.625	18 UNF-2A	RH	EXT
240		3/8	18 NGT	RH	INT
296	Bullet Nipple	0.803	14 UNS-2B	RH	INT
300	Conical Nipple	0.825	14 NGO	RH	EXT
320	Flat Nipple	0.825	14 NGO	RH	EXT
326	Small Round Nipple	0.825	14 NGO	RH	EXT
330	Flat Nipple	0.825	14 NGO	LH	EXT
346	Large Round Nipple	0.825	14 NGO	RH	EXT
347	Long Round Nipple	0.825	14 NGO	RH	EXT
350	Round Nipple	0.825	14 NGO	LH	EXT
500	Bullet Nipple	0.885	14 NGO	RH	INT
510		0.885	14 NGO	LH	INT
540		0.903	14 NGO	RH	EXT
580		0.965	14 NGO	RH	INT
590		0.965	14 NGO	LH	INT
660	Face Washer	1.03	14 NGO	RH	EXT
670	Face Washer	1.03	14 NGO	LH	EXT
677	Round Nipple	1.03	14 NGO	LH	EXT
678	Recessed Washer	1.03	14 NGO	LH	EXT
679	Tipped Nipple	1.03	14 NGO	LH	EXT
680		1.045	14 NGO	RH	INT
695		1.045	14 NGO	LH	INT
703		1.125	14 NGO	LH	INT
705	Face Washer	1.125	14 UNS-2A	RH	EXT
973	Pin Indexed Yoke		Pins 11 - 24		

Sources: [6, 7, 52]

Metering Gases



Charging or metering gases to reactor vessels must be undertaken carefully, with a well-planned setup to accomplish the transfer safely and accurately. The diagram at left depicts a typical setup. Remember that cylinders must always be secured if the cap is off.

Ensure that all materials are compatible with the gases being used, and that the system is designed to withstand the intended pressures. Ideally, the system should be designed to handle the full cylinder pressure in the event of a regulator failure. Include a valve to relieve residual pressure before disassembly. Pay attention to where this relief line is vented, especially when using corrosive or flammable gases. A check valve is recommended if there is any possibility of water or other substances backing up into the regulator or gas cylinder, particularly when charging subsurface via a sparge or dip tube.

Before introducing product, always leak test any new installations, or those that have undergone any modifica-

tions, using an inert gas such as nitrogen or argon. Also use inert gas to flush the lines of air, if using air-sensitive compounds, and to flush the lines of product prior to disassembly.

The toploader balance, rotameter or flowmeter are all convenient ways to monitor the progress of the transfer. Make sure that the device used provides the necessary degree of accuracy, and in the case of rotameters or flowmeters, that the materials and pressure ratings are acceptable for the application. Note that the temperature of liquified gas cylinders may drop during discharge, affecting the accuracy of any of the above monitoring devices.

Heating Gas Cylinders Safely

As mentioned above, the temperature, and therefore the pressure, of a liquified gas cylinder drops as it is discharged. Maintaining constant gas flow under these conditions is difficult. Cylinders may be warmed to alleviate this problem, but no part of a gas cylinder should ever be heated to a temperature above 125°F (52°C). Likewise, warm water baths, commonly used for this purpose, should be avoided because of the possibility of eventual corrosion and cylinder rupture.

The only safe way to heat a gas cylinder is with the use of an approved thermostatically controlled cylinder heating system. These are available from several sources, such as Therman Engineering (www.thermon.com). Such units are designed to operate in safe or hazardous zones and provide safe, even heating of the cylinder and its contents.

The maximum theoretical continuous steady-state flowrate can be estimated if the enthalpy of vaporization of the product and the heat output of the device are known. The example below illustrates this.

Liquified Gas Cylinder Discharge Example

Estimate the amount of HCl gas which can be discharged from a cylinder, using a 1500 Watt heater with an efficiency of 90%. From Page 7-12, the molecular weight of HCl is 35.6, and ΔH_{vap} is 16.2 kJ/g-mole. The following relationship is used (m = mass flowrate, Q = heat input):

$$m = \frac{Q \times \text{efficiency}}{\Delta H_{\text{vap}}} = 1500 \text{ W} \times 0.9 \times \frac{\text{J}}{\text{sec-W}} \times \frac{3600 \text{ sec}}{\text{hr}} \times \frac{\text{mole}}{16200 \text{ J}} \times \frac{0.0365 \text{ kg}}{\text{mole}} = \frac{10.9 \text{ kg}}{\text{hr}}$$

This calculation ignores heat-up time and heat losses to the environment. Based on [125].

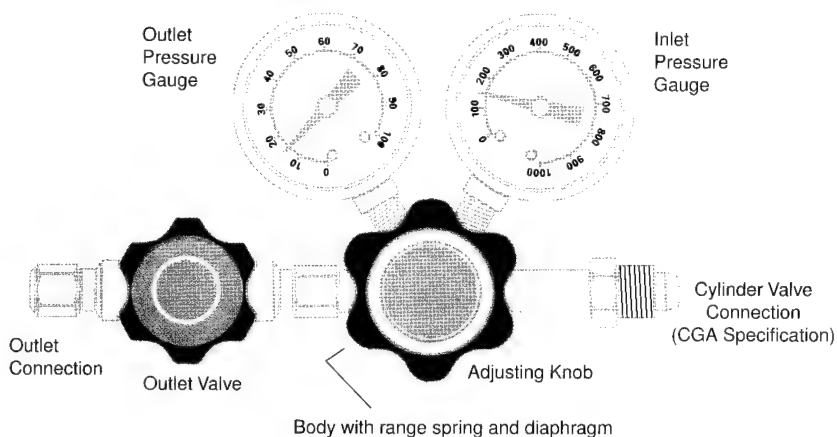
Care and Use of Gas Pressure Regulators

Regulator Selection – Regulators are specified according to the specific gas, the inlet pressure gauge required, outlet pressure gauge required, body material, type of diaphragm material (metal, rubber, etc.), number of stages (single or two stage), purge requirements if any (cross or tee purge), type of outlet connection required (hose nipple, pipe thread) and CGA inlet type. Always select the appropriate regulator for the application and CGA connection type (see page 7-6).

In the case of corrosive gases, such as chlorine, hydrogen bromide, hydrogen chloride, etc., pay special attention to the body and diaphragm materials. Monel is the recommended alloy in these cases. For more complete information on materials compatibility see Chapter 10. Most major suppliers will list the proper regulator for almost every gas, pressure and situation. If there are questions about the proper regulator to use, contact your vendor.

Single Stage or Two-Stage? – Regulators work by maintaining a trans-diaphragm pressure balance between internal cylinder pressure and the setpoint range spring. In **single stage** regulators, delivery pressure increases as cylinder pressure decays. Frequent adjustments may be required to maintain constant delivery pressure (this is not the case for liquefied gases). Where constant delivery pressure is critical, **two-stage** regulators should be used. These provide a more constant delivery pressure by establishing an intermediate pressure that varies little as cylinder pressure decreases.

A Typical Gas Pressure Regulator



Safe Regulator Use – Before use, check the regulator label. Inspect the regulator for evidence of damage, contamination or wear. Ensure that the CGA connection is appropriate for the intended use and that the outlet pressure gauge is suitable for the cylinder or source pressure.

Inspect the cylinder valve connection for damage or contamination. Clear any foreign material before attempting to attach the regulator. Attach the regulator and tighten the inlet nut. Do not overtighten. Close the regulator (turn the adjusting knob to the full counterclockwise position) and then slowly open the cylinder valve. Inspect the system for leaks. Always pressurize a regulator slowly, while standing with the regulator valve between you and the regulator.

When removing the regulator from service, always close the cylinder valve first, then vent the gas in the regulator body by turning the control knob fully clockwise. This is especially important in two-stage regulators that can trap high pressure gas in the first stage. In the case of corrosive or toxic gases, ensure that the regulator has been purged with inert gas before venting to the open atmosphere. Close the regulator by turning the control knob fully counterclockwise. Disconnect any other equipment or lines, then remove the regulator from the cylinder. Replace the cylinder outlet seal and the valve cap.

If the regulator is to be out of use for an extended period, protect the inlet and outlet from dirt, contamination or mechanical damage. Always keep regulators clean. Inspect regulators periodically for wear and leaks. Replace worn out regulators before they fail.

Never exchange the outlet pressure gauge for one of lower pressure. The gauge could rupture if the adjusting knob is turned too far. Never swap inlet fittings or use the regulator for a gas other than that for which it was intended. Never lubricate a regulator or use pipe dopes when connecting to a cylinder or other high pressure system. Provide back-pressure check valves when using high pressure equipment to prevent backflow to the cylinder and damage to the regulator. Never reverse flow through a regulator or depend on it to act as a check valve. It will not perform this function.

Physical Properties of Gases

Table Notes – The tables below list some important properties of a number of common gases and cryogenic liquids. The key characteristics column indicates any significant hazards associated with the use of the gas and its physical form (i.e. liquefied gas) as sold in cylinders. The vapor pressure is reported in psia at 70°F (21°C). The specific gravity is reported relative to that of air at 70°F (21°C) and 1 atmosphere pressure (at these conditions, the density of air is 0.075 lb/ft³ or 1.203 kg/m³ or 0.1203g/ml). The flammability limits in air indicate the range of ignitable concentrations, in volume %, in air at 1 atmosphere pressure. Viscosity is also reported with respect to air at 68°F (20°C) and 1 atmosphere (at these conditions, air has a viscosity of 0.0186 cP). Unlike liquids, the viscosity of gases *increases* with temperature. The increase is roughly linear – for every 35°C rise in temperature, viscosity increases about 10-20%. Finally, the enthalpy of vaporization is given for liquefied gases in kJ/gmole at their normal boiling point. To convert any of these quantities to other units, see the conversion factors in Chapter 11. Sources [6, 7, 110, 154, 176, 195, 237]

Properties of Some Common Gases

Gas	Mol Wt.	Formula	Key Characteristics	Vapor Pressure (psia)	Specific Gravity (air=1)	Flammability Limits in air(%)	Viscosity at 20°C (Air=1)	DHvap kJ/gmole at NBP
Acetylene	26.04	C ₂ H ₂	flammable gas	650	0.906	2.5 - 80	0.56	-
Ammonia	17.03	NH ₃	toxic, liquified gas	129	0.594	16 - 25	0.54	23.3
Argon	39.95	Ar	inert gas	-	1.380	NF	1.23	-
Boron Trichloride	117.17	BF ₃	toxic, corrosive, liquified gas	19	4.100	NF	-	23.8
Boron Trifluoride	67.82	BCl ₃	toxic, corrosive gas	-	2.380	NF	0.92	-
Bromotrifluoromethane (Freon 13B1)	148.91	CBrF ₃	liquified gas	208	5.310	NF	-	-
1,3-Butadiene	54.09	C ₄ H ₆	flammable, liquified gas	36	1.878	2.0 - 11.5	-	22.5
Butane	58.12	C ₄ H ₁₀	flammable, liquified gas	31	2.110	1.8 - 8.4	0.40	22.4
Butene-1	56.11	C ₄ H ₈	flammable, liquified gas	37	2.032	1.6 - 10	0.46	22.1
Carbon Dioxide	44.01	CO ₂	compressed gas	845	1.522	NF	0.81	-
Carbon Monoxide	28.01	CO	flammable gas	-	0.967	12.5 - 74	0.96	-
Chlorine	70.91	Cl ₂	toxic, corrosive, liquified gas	100	2.473	oxidizer	0.72	20.4
Chlorodifluoromethane (Freon 22)	86.47	CHClF ₂	liquified gas	138	3.110	NF	0.65	20.2
Chlorotrifluoromethane (Freon 13)	104.46	CClF ₃	liquified gas	473	3.610	NF	-	15.8
cis-2-Butene	56.11	C ₄ H ₈	flammable, liquified gas	28	1.997	1.6 - 9.7	-	23.3
Dichlorodifluoromethane (Freon 12)	120.91	CCl ₂ F ₂	liquified gas	85	4.200	NF	0.65	20.1
1,2-Dichlorotetrafluoroethane (Freon 114)	170.93	C ₂ Cl ₂ F ₄	liquified gas	27.6	5.930	NF	0.68	23.3
Dimethyl Ether	46.07	(CH ₃) ₂ O	flammable, liquified gas	77	1.621	3.4 - 27	0.50	21.5
Dimethylamine	45.08	(CH ₃) ₂ NH	flamm., alkaline, liquif. gas	26	1.557	2.8 - 14.4	-	26.4
Ethane	30.07	C ₂ H ₆	flammable, liquified gas	558	1.048	3.0 - 12.4	0.51	14.7
Ethyl Chloride	64.52	C ₂ H ₅ Cl	flammable, liquified gas	20	2.230	3.8 - 15.4	0.53	24.7
Ethylene	28.05	C ₂ H ₄	flammable gas	-	0.974	2.7 - 36	0.56	-
Ethylene Oxide	44.05	C ₂ H ₄ O	toxic, flammable, liquified gas	22	1.490	3.1 - 100	-	-
Fluoroform (Freon 23)	70.01	CHF ₃	liquified gas	650	2.417	NF	-	-
Helium	4.00	He	inert gas	-	0.138	NF	1.08	-
Hexafluoroethane (Freon 116)	138.01	C ₂ F ₆	liquified gas	445	4.823	NF	-	16.2
Hydrogen	2.02	H ₂	highly flammable gas	-	0.070	4.0 - 75	0.48	-

See Table Notes above.

Properties of Some Common Gases (continued)

Gas	Mol Wt.	Formula	Key Characteristics	Vapor Pressure (psia)	Specific Gravity (air=1)	Flammability Limits in air(%)	Viscosity at 20°C (Air=1)	ΔH_{vap} kJ/gmole at NBP
Hydrogen Bromide	80.92	HBr	toxic, corrosive, liquified gas	335	2.812	NF	0.99	17.4
Hydrogen Chloride	36.46	HCl	toxic, corrosive, liquified gas	628	1.267	NF	0.78	16.2
Hydrogen Sulfide	34.08	H ₂ S	toxic, flammable, liquified gas	267	1.188	4.0 - 44	0.68	18.7
Isobutane	58.12	C ₄ H ₁₀	flammable, liquified gas	45	2.064	1.8 - 8.4	0.41	21.3
Isobutylene	56.11	C ₄ H ₈	flammable, liquified gas	39	1.947	1.8 - 9.6	0.42	-
Krypton	83.80	Kr	inert gas	-	2.890	NF	1.38	-
Methane	16.04	CH ₄	flammable gas	-	0.555	5.0 - 15	0.6	-
Methyl Bromide	94.94	CH ₃ Br	toxic, flammable, liquified gas	28	3.355	10 - 16	0.72	23.9
Methyl Chloride	50.49	CH ₃ Cl	liquified gas	73	1.784	8.1 - 17	0.57	21.4
Methyl Mercaptan	48.11	CH ₃ SH	liquified gas	30	1.660	3.9 - 21.8	-	-
Monomethylamine	31.06	CH ₃ NH ₂	toxic, flamm., alkaline, liq. gas	17	1.610	4.9 - 20	-	25.6
Neon	20.18	Ne	inert gas	-	0.696	NF	1.73	-
Nitric Oxide	30.01	NO	toxic gas	-	1.036	NF	1.03	-
Nitrogen	28.01	N ₂	compressed gas	-	0.967	NF	0.96	-
Nitrogen Dioxide	46.01	NO ₂	toxic, corrosive, liquified gas	14.7	2.620	oxidizer	-	-
Nitrous Oxide	44.01	N ₂ O	oxidizing gas	760	1.530	oxidizer	0.81	-
Oxygen	32.00	O ₂	oxidizing gas	-	1.105	oxidizer	1.12	-
Perfluoropropane	188.02	C ₃ F ₈	liquified gas	115	6.690	NF	-	-
Phosgene	98.92	COCl ₂	toxic, corrosive, liquified gas	-	-	NF	-	24.2
Phosphine	34.00	PH ₃	highly toxic, flammable gas	-	1.180	pyrophoric	0.62	-
Propane	44.10	C ₃ H ₈	flammable, liquified gas	124	1.550	2.2 - 9.5	0.45	19.0
Propylene	42.08	C ₃ H ₆	flammable, liquified gas	151	1.480	1.9 - 11.1	0.45	18.4
Silane	32.12	SiH ₄	pyrophoric gas	-	1.110	pyrophoric	0.62	-
Silicon Tetrafluoride	104.10	SiF ₄	toxic, corrosive gas	-	3.604	NF	-	-
Sulfur Dioxide	64.06	SO ₂	toxic, liquified gas	49	2.263	NF	0.69	24.9
Sulfur Hexafluoride	146.05	SF ₆	liquified gas	335	5.114	NF	-	-
Tetrafluoromethane (Freon 14)	88.01	CF ₄	compressed gas	-	3.050	NF	0.92	-
trans-2-Butene	56.11	C ₄ H ₈	flammable, liquified gas	30	1.997	1.6 - 9.7	-	22.7
Trimethylamine	59.11	(CH ₃) ₃ N	toxic, flammable gas	28	2.087	2.0 - 11.6	-	-
Xenon	131.30	Xe	inert gas	-	4.553	NF	1.25	-

See Table Notes previous page.

Properties of Some Cryogenic Liquids

Liquid	Mol Wt.	Formula	Boiling Pt °C at 1 atm	Boiling Pt °K at 1 atm	Liquid Density g/cm ³ at NBP	ΔH_{vap} at NBP kJ/g-mole	Critical T °C	Critical P atm
Air	28.69	-	-194.5	78.7	0.88	5.70	-140.65	37.3
Argon	39.95	Ar	-185.9	87.3	1.40	6.43	-122.49	48.1
Helium	4.00	He	-268.9	4.2	0.12	0.08	246.35	2.3
Hydrogen	2.02	H ₂	-252.9	20.3	0.07	0.90	-240.17	12.8
Krypton	83.80	Kr	-153.2	119.9	2.42	9.08	-63.75	54.5
Methane	16.04	CH ₄	-161.5	111.7	0.42	8.19	-82.59	45.5
Neon	20.18	Ne	-246.1	27.1	1.20	1.71	-228.75	27.3
Nitrogen	28.01	N ₂	-195.8	77.4	0.81	5.57	-146.95	33.6
Oxygen	32.00	O ₂	-183.0	90.2	1.14	6.82	-118.57	49.9
Xenon	131.29	Xe	-108.1	165.1	2.95	12.62	16.58	57.8

Gas Leak Detection

Gas cylinder setups, especially for hazardous or flammable gases, should always be tested for leaks prior to use. Usually the most convenient way to do this is to pressurize the system with an inert gas and spray the connections with soapy water or a commercial leak detector such as “Snoop”. Check the compatibility of any leak detector with the particular gas and materials being used.

In the case of particularly flammable or explosive gases such as hydrogen, extreme care should be taken to prevent leaks. The use of a specifically designed electronic monitoring systems is strongly recommended. Personal and stationary monitoring systems are available for most hazardous or combustible gases. Discuss the details with your gas supplier.

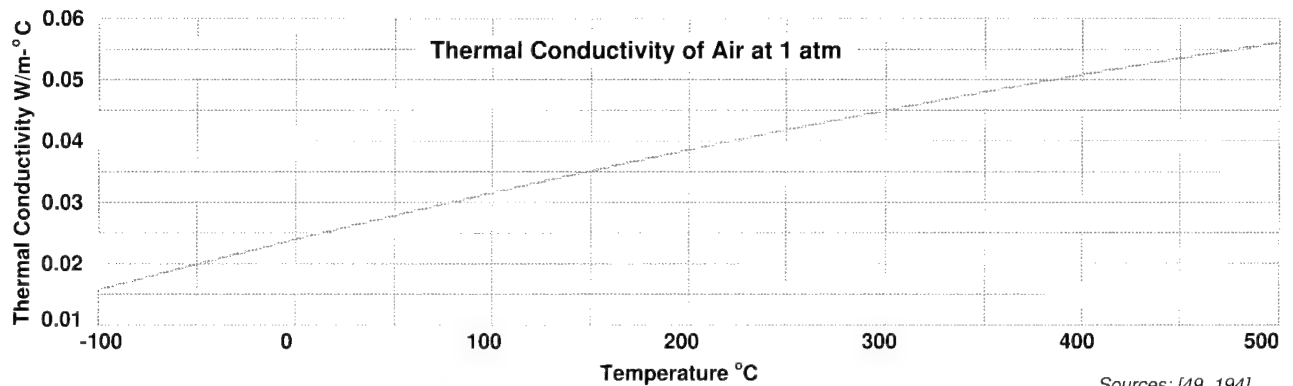
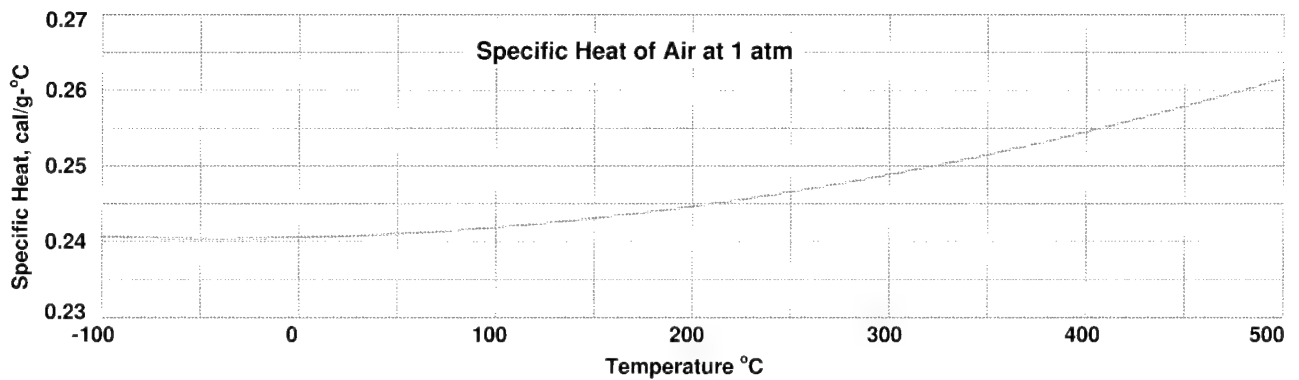
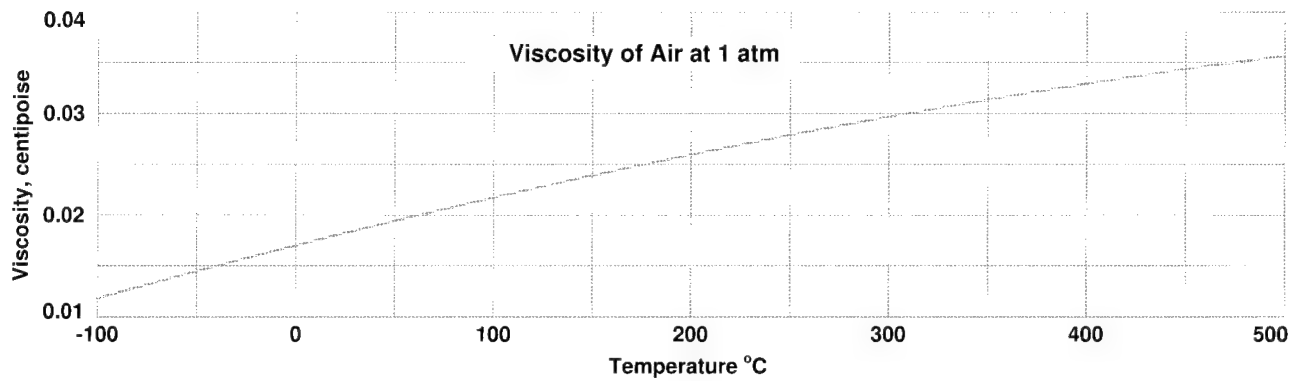
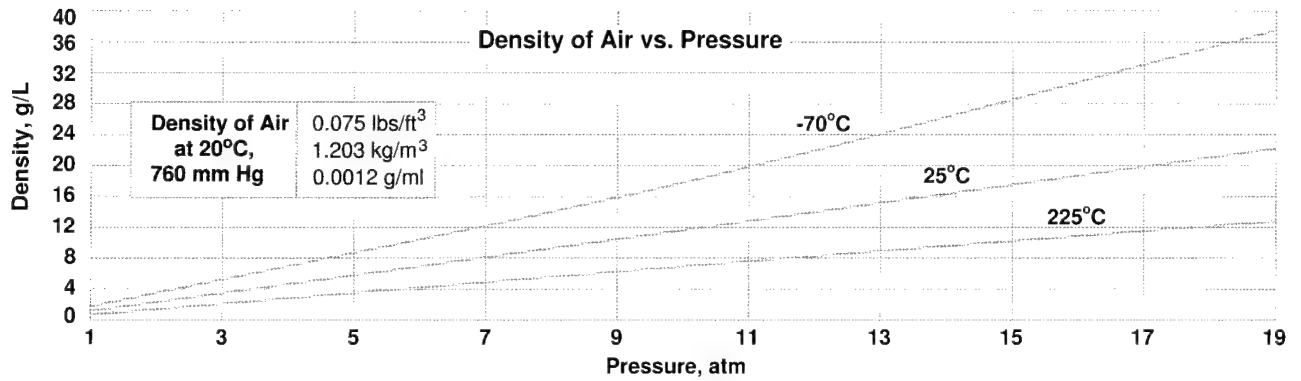
In addition, a number of convenient methods can help you to detect leaks of certain gases in a pinch. Some of these are listed below. Many of these tests use substances that are themselves harmful, and should be used with caution and only in properly ventilated areas.

Quick Gas Detection Techniques

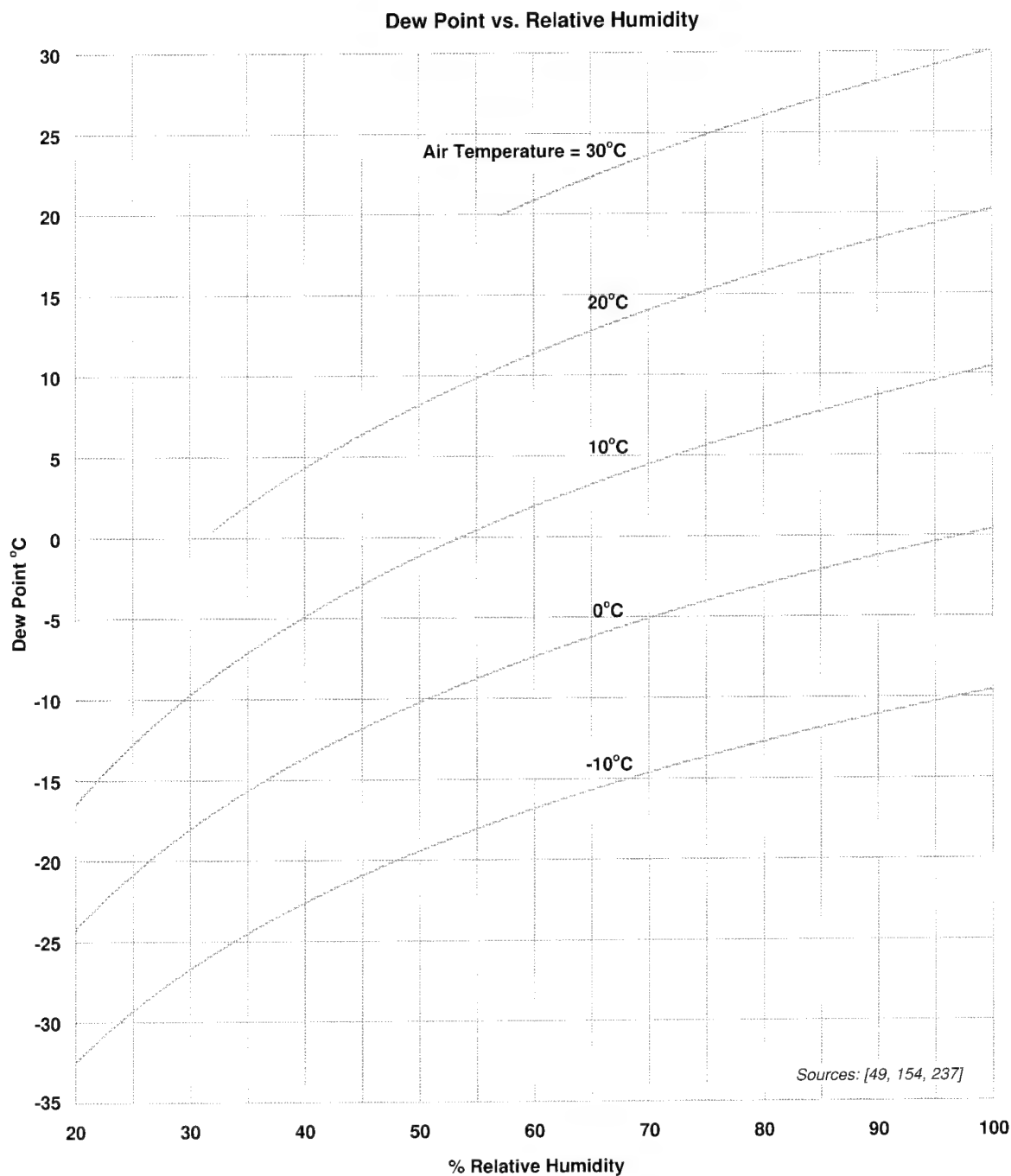
Gas	Test Material	Method
HCl	Aqueous Ammonium Hydroxide	Vapors form white fumes near point of leak.
	Mixture of solid Na_2CO_3 and NH_4Cl	Generates NH_3 and forms white fumes near point of leak.
HBr	Aqueous Ammonium Hydroxide	Vapors form white fumes near point of leak.
	Mixture of solid Na_2CO_3 and NH_4Cl	Generates NH_3 and forms white fumes near point of leak.
	Cu(II) acetate / benzidine acetate paper	Paper turns blue.
HF	Zirconium-alizarin paper	Paper moistened with 50% acetic acid turns yellow. Paper is prepared by soaking filter paper in 5% $\text{Zr(NO}_3)_4$ in 5% aq HCl, then in 2% aq Na alizarin sulfonate.
HI	Aqueous Ammonium Hydroxide	Vapors form white fumes near point of leak.
	Mixture of solid Na_2CO_3 and NH_4Cl	Generates NH_3 and forms white fumes near point of leak.
	Cu(II) acetate / benzidine acetate paper	Paper turns blue.
HCN	Cu(II) acetate / benzidine acetate paper	Paper turns blue.
H₂S	Lead acetate paper	Wet paper turns black.
O₃	Silver foil	Foil turns black.
	MnCl_2 paper	Paper turns brown.
Cl₂	Starch-iodine paper	Paper turns blue.
Br₂	Starch-iodine paper	Paper turns blue.
	Fluorescein paper	Paper turns red.
I₂	Starch-iodine paper	Paper turns blue.
	Fluorescein paper	Paper turns red.
NH₃	Concentrated HCl	Vapors form white fumes near point of leak.
SO₂	Starch-iodine paper	Paper decolorizes.

Adapted from [72, 110]

Properties of Air



Sources: [49, 194]



The chart above shows the dew point vs. relative humidity for several air temperatures at atmospheric pressure. The dew point is the temperature at which moisture just begins to condense on cold surfaces. It is a function of both the air temperature and the % relative humidity. At 100% relative humidity, the dewpoint equals the air temperature. By way of example, at an air temperature of 20°C and a relative humidity of 75%, moisture will begin to condense on any surfaces that are colder than the 15°C dew point.

Compressed Air Systems

The selection or design of a compressed air system requires consideration of a number of important factors including expected load and capacity needs, air quality requirements, inlet and discharge piping, distribution system characteristics, etc. Engineering details and specifications can be obtained from any reputable dealer in compressed air systems, but here are the main things to keep in mind:

Compressor Type – The type of compressor selected depends on pressure and flowrate requirements as well as some other considerations described below. The figure at the bottom of the page shows the five major compressor types. Centrifugal compressors are oil-free units that operate in several stages at very high rpm and are typically large in size (>2000 CFM). Flexible diaphragm units are designed to provide relatively low pressures only. Reciprocating, or piston-type compressors, are generally not well-suited for continuous operation and should be sized to operate on a 60-70% duty cycle; single stage units provide air at 95-125 psi. Rotary screw compressors are oil-flooded positive displacement units that generate higher pressures (~150 psi) than rotary vane or piston-type. Rotary or sliding vane type compressors are positive displacement units that operate in the 100-110 psi range.

CFM Requirements – The expected air usage in cubic feet/min depends on the tools or equipment you will use (see the chart on the opposite page for a general idea of typical air usages). The system should be oversized to run at a duty cycle of about 60%. Exceeding a unit's rated duty cycle will shorten its life.

Pressure Requirements – Again, this is based on the equipment you plan on using. Excess pressure should always be provided to account for losses in filters, dryers and piping. Note that compressor outlet pressure is usually listed in psig, or lbs/sq.in.-gauge (thus atmospheric pressure = 0 psig).

Duty Cycle – This represents the amount of compressed air used as a percentage of the maximum amount available over time. A unit that operates continuously just to match the demand for air has a 100% duty cycle, which is not recommended for reciprocating compressors and some other types. Setting an appropriate duty cycle requires proper sizing of the compressor and the air reservoir and careful consideration of all air requirements, large and small.

Horsepower Requirement – This is directly related to CFM and pressure requirements. The chart of theoretical horsepower requirements on the following page provides a very general guideline.

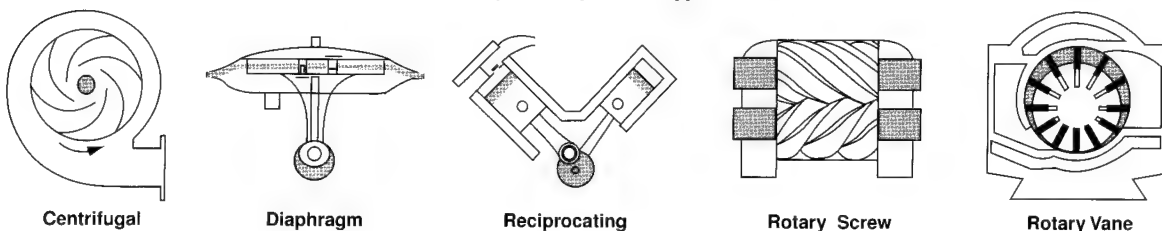
Primary Power Source – The electrical system must be able to meet requirements of compressor running and starting loads. 115 V is common for smaller, portable compressors, 230V or 460 V for higher horsepower machines (motors are usually 3-phase over 5HP). These larger units usually require a separate motor starter and are permanently hard-wired in place. Also keep in mind that air compressors operate most efficiently under full load.

Space Limitations – The location is based on a number of factors, such as cooling method (air- or water-cooled), noise considerations and whether there is any need to bolster the foundation depending on the size and type of unit.

Aftercoolers and Separators – The air discharged from compressors is hot and usually contains water, oil, and other contaminants. Heat exchangers (aftercoolers) are normally installed to reduce the air temperature and knock out the majority of the water vapor. These may be water- or air-cooled. It may also be necessary to install a moisture separator to collect the condensed water vapor and direct it to a drain valve, or an oil trap.

Air Receiver – The capacity of the air receiver or reservoir again depends on expected usage rates and duty cycle

Principle Compressor Types



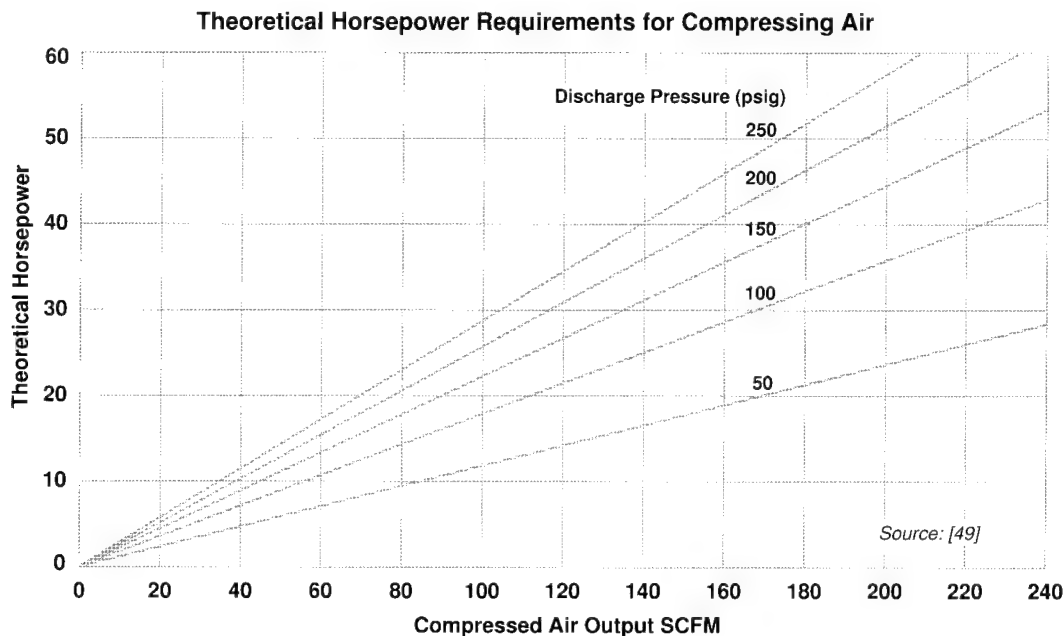
requirements. A rough estimate of required receiver size can be made from the chart on page 7-18.

Air Treatment Systems – The required air quality will determine the need for any one of a number of treatment systems, including particulate filters, oil separators, and air dryers. Several types of refrigerated or desiccant-type air dryers are in common use. The pressure drop incurred by these devices must be taken into account in compressor sizing. If different air quality needs are expected at different points of use, it may be more economical to treat smaller amounts of air for a particular use than to treat the entire air supply.

Drain Valves – To remove condensed water vapor from the air reservoir, some type of automated drain valve must be included. These may be float valves or electronic solenoid types. Note that the condensate from compressed air systems (which contains lubricants and other contaminants) must be disposed of in accordance with the appropriate hazardous waste disposal guidelines.

Point-of-Use Components – These include directional control valves, in-line particulates filters, pressure regulators, oil separators and lubricators, which must all meet the necessary safety as well as process requirements.

Air Distribution System – To ensure safe and efficient operation, the air distribution system must be designed carefully. Some considerations include the material, sizing and slope of piping, the location of drain legs, service valves and secondary reservoirs, and the need for flexible connections. Discuss the details with a reputable dealer or contractor.

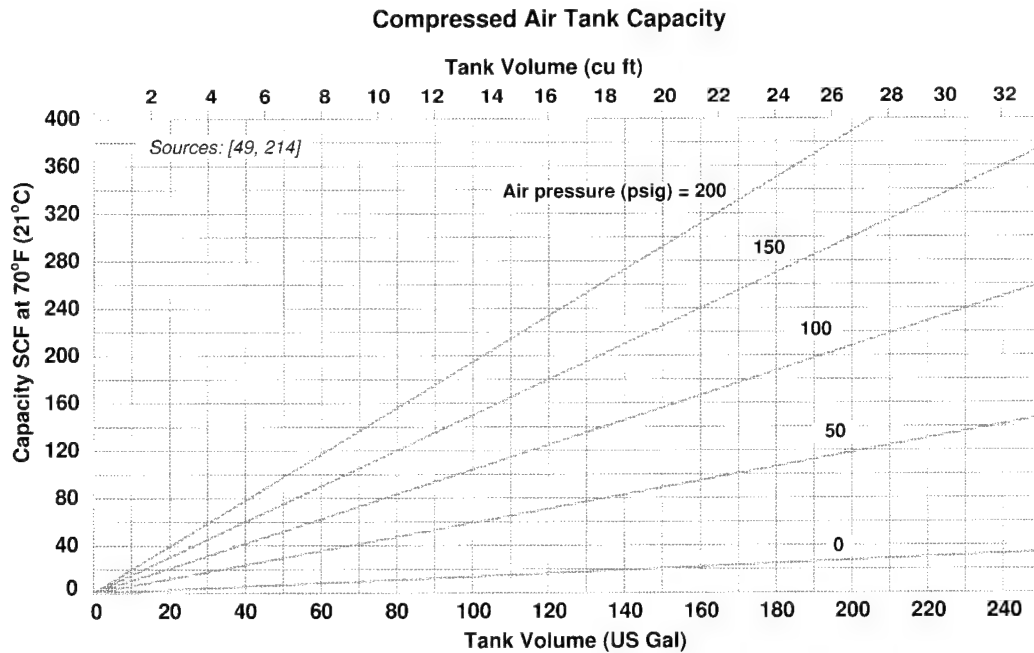


The chart above shows the theoretical horsepower requirements to compress air at a given flowrate in standard cubic feet per minute at sea level. Actual efficiencies of modern single and multiple stage compressors fall in the range of 70 to 80%, and thus actual compressor horsepower requirements should be increased accordingly.

Some Typical Compressed Air Usage Rates

Device	Air Consumption Rate SCFM	Air pressure, psig
Diaphragm pump, 25 gpm at 200 ft	25	100
Diaphragm pump, 80 gpm at 50 ft	60	50
Typical sump pump	50	60
1/2 hp air-driven mixer-motor	25	80
1 hp air-driven mixer-motor	40	90

Sources: [49, 214]

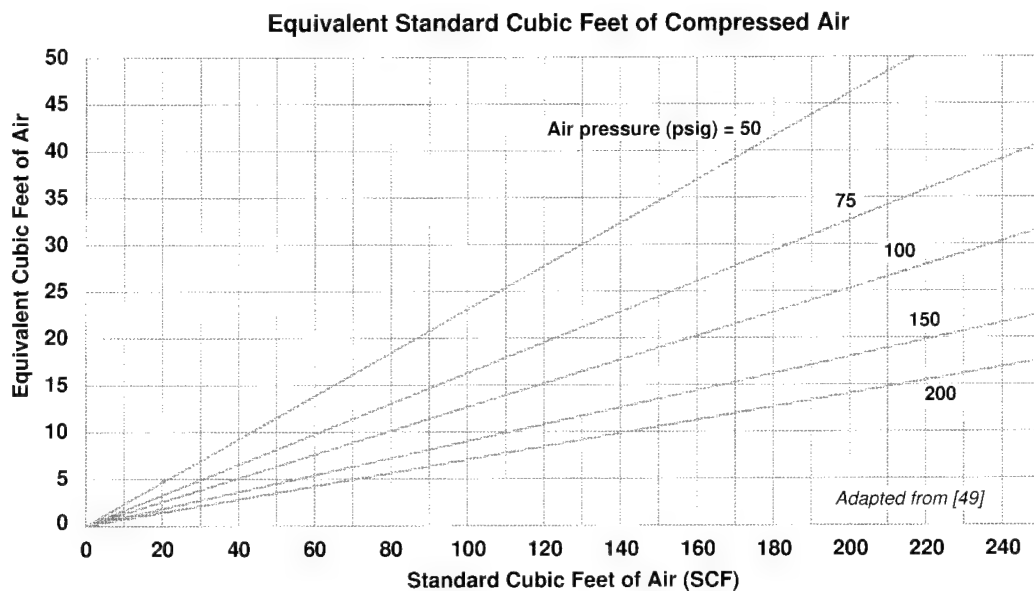


The chart above shows the capacity of compressed air receiver tanks and is provided as a rough guide for sizing receivers. The correct receiver size depends on the expected usage and supply rates. The following relationship will enable you to estimate if a receiver is properly sized for the application.

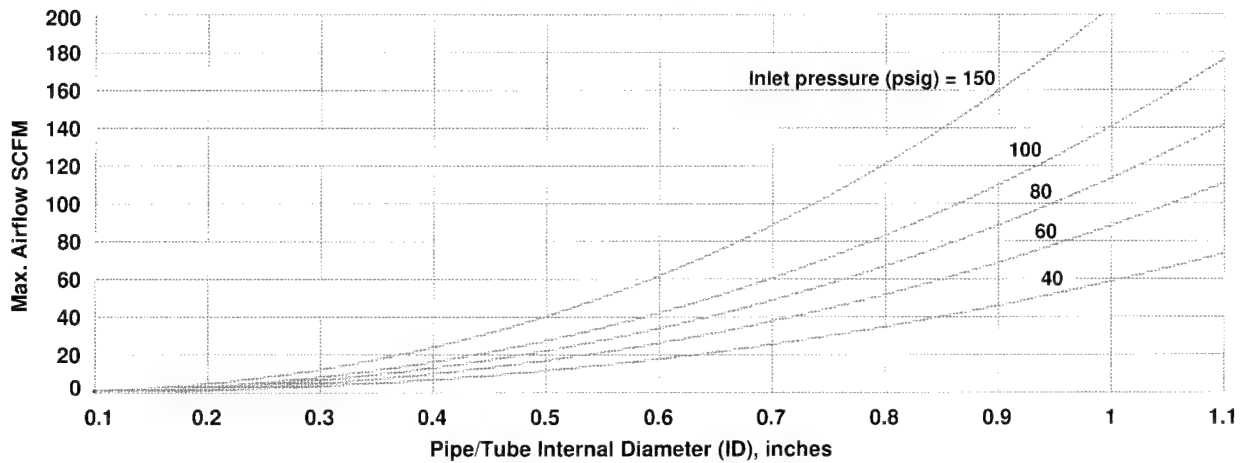
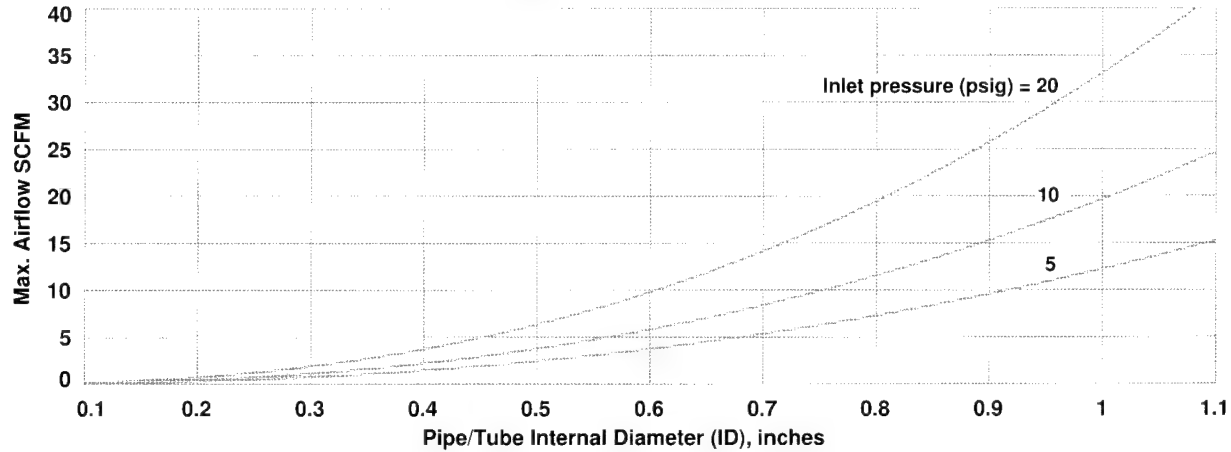
$$\text{Time to empty a compressed air receiver (min)} = \frac{V \times (P_o - P_f)}{(C - S) \times 14.7}$$

This allows calculation of the time (in minutes) required to reduce the pressure in the vessel from the starting pressure P_o to the final pressure P_f (both in psig) for a tank of given volume V (ft^3) based on an air consumption rate C and a compressor air supply rate S (both in SCFM).

The chart below shows the equivalent SCF of compressed air at various pressures.



Air Flow Through Pipes and Tubes vs. Pressure

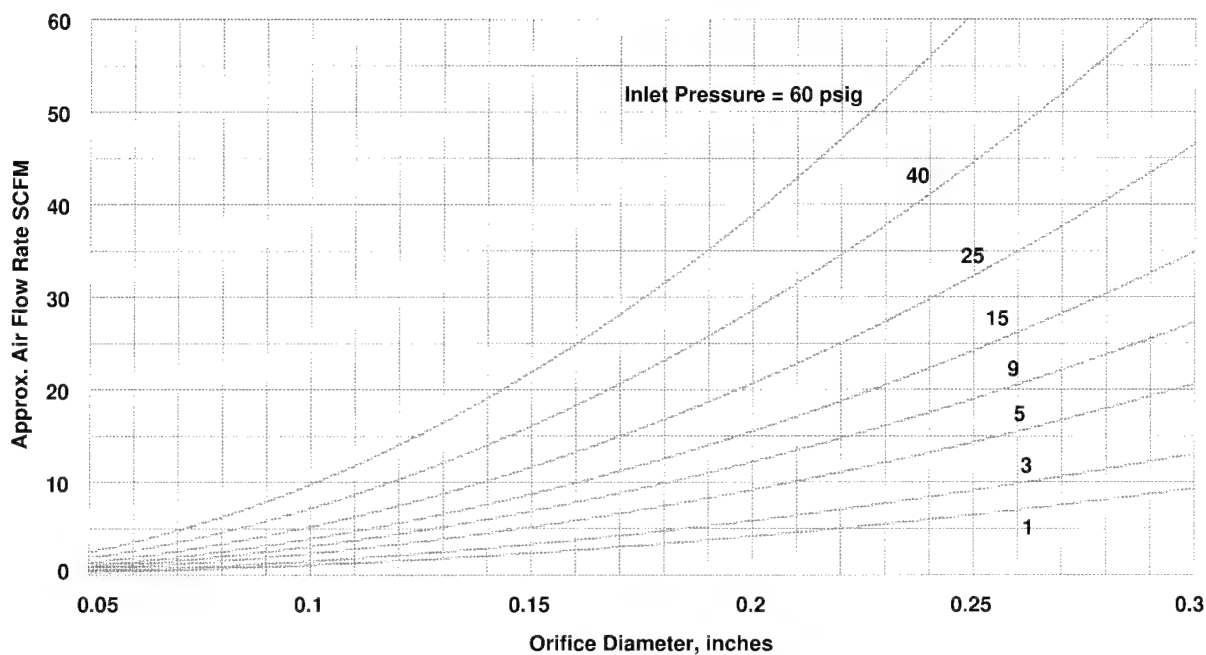
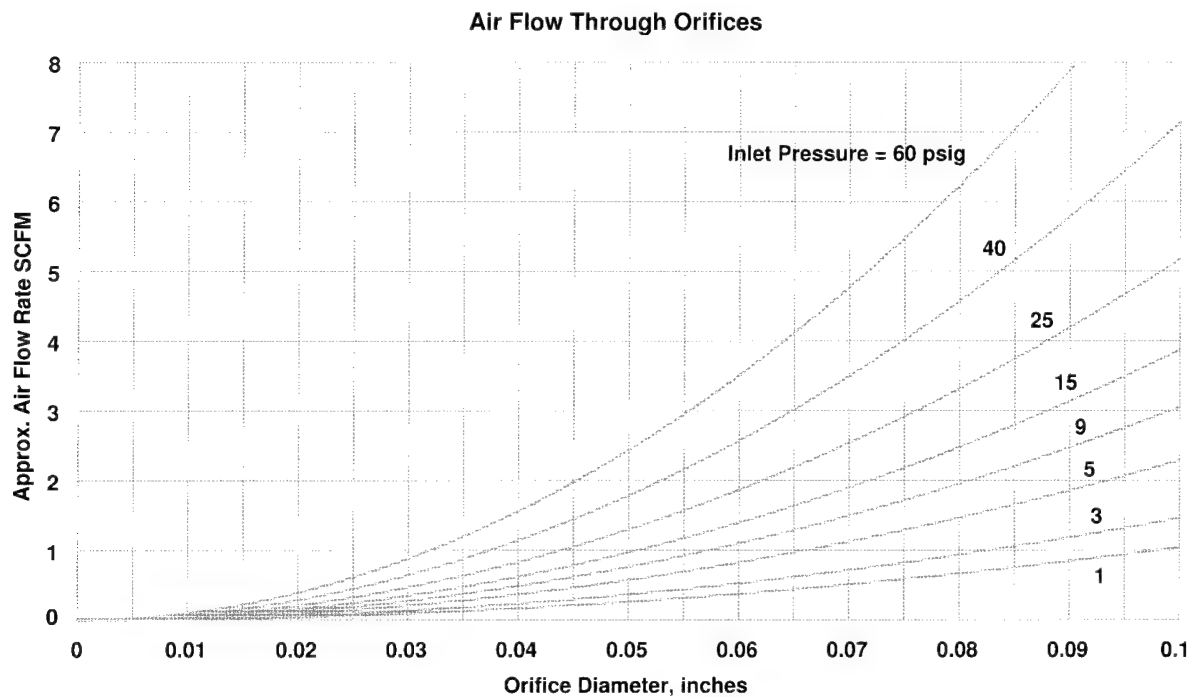


The above charts can be used as rough guides when sizing piping for compressed air systems. They represent the maximum recommended air flow in standard cubic feet per minute per 100 linear foot pipe run. The numbers are based on a pressure drop of about 10% of applied pressure for pipes with ID < 0.6" and a pressure drop of about 5% of applied pressure for pipes with ID > 0.6". Pipe and tubing comes in many materials, wall thicknesses and schedules. Always ensure that the particular components used are rated for the maximum system pressure. Some representative pipe and tube internal dimensions are given on pages 3-14 to 3-16. Source [49].

Pressure Drop Through Standard Fittings for Air, Steam or Gases
(as equivalent length of straight pipe in feet)

Nominal Schedule 40 pipe size	Gate valve	Globe valve	Run of standard tee	Run of tee reduced 50%	Standard elbow	Side Outlet of tee	Tight return bend
1/2"	0.4	17.0	0.6	1.6	0.6	3.1	3.5
3/4"	0.5	23.0	0.8	2.1	0.8	4.1	4.6
1"	0.6	29.0	1.0	2.6	1.0	5.2	5.8
1-1/2"	0.9	45.0	1.6	4.0	1.6	8.0	9.0

See page 3-24 for pressure drop of liquids in pipes and fittings.



The charts above represent the approximate flow of air through a regular orifice of the internal diameter shown at the inlet pressure shown when discharged to atmospheric pressure. Flow through orifices can be affected by many things, including the degree of wear of the edges of the orifice. These graphs are based on an orifice with reasonably sharp edges (coefficient of flow, $C = 0.9$). Source [49].

Vacuum Systems

Vacuum is widely used in the CPI for distillation, purging and charging operations, and drying. Therefore, equipment for generating vacuum is available in many types, styles and sizes. Extremely high vacuum (less than 1 mm Hg) is often used in the laboratory, but for general service in the plant, about 50-100 mm Hg (~2-4 in. Hg) is typical.

The most common class of vacuum pumps are the oil-filled models, which rely on a reservoir of pump oil for sealing and lubrication. Various oils are available for different applications and levels of vacuum. Common oil-sealed pumps include **rotary piston** and **rotary vane** types. These are usually the first choices for plant central vacuum systems. They are similar in design to their liquid-pumping counterparts (see page 3-10). Where process contamination by oil could be an issue, **dry screw pumps** are a good alternative. With these, the need for oil or another service liquid is eliminated.

Liquid ring pumps are a type of liquid-sealed pump popular in small industrial installations. Single stage models are sized to over 1000 CFM and can produce about 100 mm Hg vacuum. They rely on a supply of fresh sealant fluid or “service liquid”, usually water, which continually flows through the pump to drain. The design minimizes wear and obviates the need for lubrication, but can consume considerable amounts of water. If the water or sealant will be recycled, a separate water pump and heat exchanger are required to remove built-up heat. However, these are simple, very reliable, low maintenance pumps. Other common types of water-sealed pumps include **liquid jet pumps**, and combination **liquid ring-booster** or **liquid ring-ejector** pumps, all of which are capable of producing deeper vacuums. **Steam jets**, and the lowly but reliable water **aspirator**, which use venturi nozzles to generate vacuum, are also commonly found.

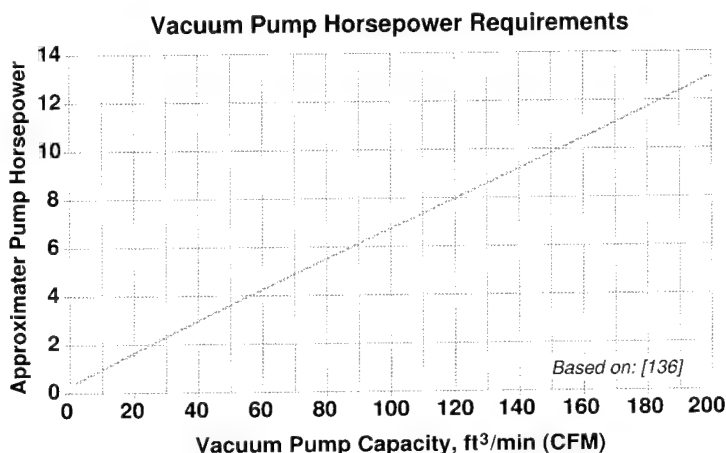
Capacity – Vacuum pump capacity is measured in “CFM”, which implies SCFM (standard ft³/min). This is the amount of air a pump will move per minute at 1 atm. As system pressure drops, so does air density, and thus the pump actually moves less and less air as it evacuates the system. A pump-down factor, F , can be calculated that relates pump-down time to final system pressure. Equations for F (valid down to 1 mm Hg) and for determining required pump capacity for a given system volume are given below [136]:

$$F = 8.6 - 1.3 \ln (P) \quad \text{for } P \text{ in torr (mm Hg), valid down to } \sim 1 \text{ torr}$$

$$\text{Required Pump Capacity (CFM)} = F \times \frac{\text{System Volume (ft}^3\text{)}}{\text{Desired Pump-Down Time (min)}}$$

For example, to calculate the required capacity to evacuate a 200 gal (27 ft³) vessel to 100 mm Hg in 5 minutes, use the first equation to determine that $F = 2.61$. The second equation then gives the required capacity as 14.1 CFM. This, of course, assumes ideal conditions, no vapor load, and no piping constrictions. To compensate for these conditions, the pump should be sized 2-3 x this theoretical capacity. The volume of the connecting piping must also be considered. It is always best to work closely with your equipment supplier in determining the best size pump for your needs.

Horsepower increases roughly linearly with capacity. The chart below shows the approximate horsepower required for a given pump capacity, based on pooled data for several styles, including liquid ring, rotary vane and rotary piston types.



Installation – The diagram at the bottom of the page shows a typical vacuum pump setup, as might be used for service with a small reactor or product dryer. The cold trap is the point where solvent vapors are condensed and collected. Because low temperatures are required to condense solvents under vacuum, dry ice/alcohol or similar mixtures are sometimes used to cool these traps in labs or kilo-labs, but electric cryogenic units are available for larger scale. Note the inclusion of a particulates filter to protect the pump from solids, and a check valve to prevent oil from being sucked back when the pump is shut down. Vacuum pumps will not act as check valves. A knockout trap is also recommended at the exhaust to collect solvent or moisture that may pass through the pump. This does not need to be as cold as the primary cold trap, since it is condensing vapors at atmospheric pressure, but it's a good idea to have pressure and/or temperature gauges to indicate that it is working correctly. A sight or level glass will help prevent overfilling traps.

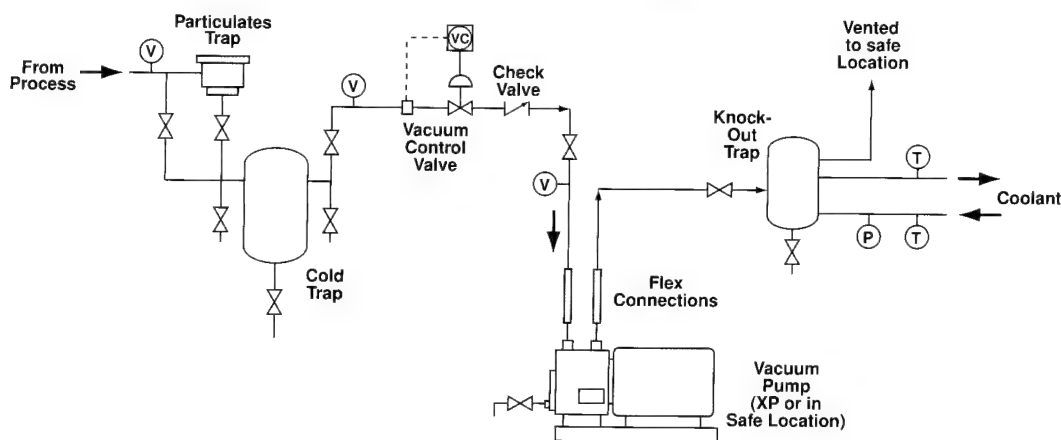
Vacuum gauges are positioned so that the operation of the pump can be checked while isolated from the rest of the system, to check the function of the vacuum control valve and to determine if the particulates filter is becoming clogged. Larger, central vacuum systems often also include a vacuum storage, or ballast tank, to handle peak loads. Installing a pump drain valve simplifies changing pump oil. Many pumps also include a built-in **gas ballast valve**. This valve provides a way to purge a controlled amount of air through the pump to remove solvent vapors or moisture. It should be used only intermittently and as necessary, however, since the pump will not achieve its full rated vacuum with it open.

Pump Maintenance – Vacuum pumps should be inspected daily to ensure sufficient oil or service fluid level and proper operation. Look for performance problems such as leaks, poor vacuum, bearing noise, vibrations or overheating. Some of these symptoms could indicate misalignment, component failure or motor overload. Oil should be changed on a regular basis according to manufacturer's maintenance schedule. Other preventive maintenance recommended by the manufacturer may include replacing seals, O-rings and gaskets.

Changing Pump Oil – The most common reason for pump failure is contaminated oil. Water contamination is quite common, and if the pump is stopped for any length of time, water can settle to the bottom and rust or corrode pump parts to the point where the pump cannot be started again. This is the reason that it is often recommended that oil-filled pumps be allowed to operate continuously. Once the pump is up to operating temperature, the water will actually be driven off as vapor. Nonetheless, water or other foreign substances in the oil will decrease the efficiency of the pump and its ability to attain its rated vacuum. Poor vacuum, cloudy oil, and solvent odors are all good indications that the oil should be changed. It is good practice to tag the pump with the time of its last oil change, or better still, to record the information, along with operating hours in the equipment log book.

Oil should be drained while the pump is warm so viscosity is low, but note that the oil can be quite hot and may give off solvent fumes. Operate the pump for a few seconds after draining to ensure removal of all oil. Use only good-quality vacuum pump oil and do not overfill. Follow any specific recommendations in the pump operating manual.

A Typical Vacuum Pump Setup



8 Chemical Data

Contents

Periodic Table of the Elements	8-2
ACIDS AND BASES	
Properties of Commercial Acids and Bases	8-3
pH of Common Acid and Base Solutions	8-3
Recipes for Dilute Acid/Base Solutions	8-4
pKa of Acids, Bases and Buffers	8-6
BUFFERS	
Properties and Preparation of Buffer Solutions	8-8
Some Useful Buffer Systems	8-9
Some NBS Standardized Buffers	8-9
pH Range of Some Acid-Base Color Indicators	8-9
SOLUBILITY	
Aqueous Solubility	8-10
General Water Solubility Rules for Inorganic Compounds	8-10
United States Pharmacopeia Definitions of Solubility	8-10
Aqueous Solubility of Selected Inorganic Compounds	8-11
Aqueous Solubility of Selected Organic Compounds	8-12
Effect of Temperature on Aqueous Solubility of Selected Compounds	8-13
Heat of Solution	8-14
DENSITY	
Solution Density	8-15
Density of Selected Aqueous Inorganic Solutions	8-16
Specific Gravity and Density Scales	8-17
MISCELLANEOUS	
Comparison of Various Concentration Scales	8-17
Grades of Chemical Purity	8-18
Drying Agents for Solvents and Solutions	8-18
CHEMICAL NOMENCLATURE	
Names of Ions	8-19
Functional Groups in Organic Chemistry	8-19
Some Common Types of Organic Reactions	8-20

Group
IA

Lanthanides

*Molecular Weight based on $^{12}\text{C} = 12$ (IUPAC 1995). Numbers in parentheses indicate the most stable isotope.

*Molecular Weight based on $^{12}\text{C} = 12$ (IUPAC 1995).

Acids and Bases

Properties of Commercial Acids and Bases

Name	Formula	Mol. Wt.	Concentration, weight %	Concentration, g/L	Concentration, moles/L	Density* (20°C)
Acetic Acid	CH ₃ COOH	60.05	99.5	1045	17.4	1.05
Butyric Acid	C ₄ H ₈ O ₂	88.11	95	912	10.3	0.96
Formic acid	HCOOH	46.03	90	1080	23.4	1.22
Hydriodic acid	HI	127.90	57	969	7.6	1.70
			47	705	5.5	1.50
Hydrobromic acid	HBr	80.91	48	720	8.9	1.52
			40	552	6.8	1.38
Hydrochloric acid	HCl	36.46	37	425	11.7	1.15
			10	105	2.9	1.05
Hydrofluoric Acid	HF	20.01	48	542	27.1	1.13
Hypophosphorous Acid	H ₃ PO ₂	66.00	50	625	9.5	1.25
Nitric Acid	HNO ₃	63.00	70	994	15.9	1.42
			67	938	14.9	1.40
Perchloric Acid	HClO ₄	100.46	70	1172	11.7	1.67
			60	923	9.2	1.54
Phosphoric Acid	H ₃ PO ₄	98.00	85	1445	14.7	1.71
Sulfuric Acid	H ₂ SO ₄	98.10	96	1766	18.0	1.84
Sulfurous Acid	H ₂ SO ₃	82.08	6	61.2	0.7	1.02
Ammonia (Ammonium Hydroxide)	NH ₃ (NH ₄ OH)	17.03 (35.05)	28 (57.6)	252 (519)	14.8	0.90
			50	757	13.5	1.52
Potassium Hydroxide	KOH	56.11	45	657	11.7	1.46
			10	109	1.9	1.09
Sodium Hydroxide	NaOH	40.00	50	763	19.2	1.53
			10	111	2.8	1.11

*For densities of acid/base solutions of other concentrations, see page 8-15.

Sources: [70, 154, 253, 259, 267]

pH of Common Acid and Base Solutions

Acid	0.001N	0.01N	0.1N	1N	Base	0.001N	0.01N	0.1N	1N
Acetic Acid	3.9	3.4	2.9	2.4	Ammonia	10.3	10.6	11.1	11.6
Arsenious Acid		5.0 (sat)			Borax	-	-	9.2	-
Benzoic Acid	-	3.1	3.0	-	Calcium Carbonate		9.4 (sat)		
Boric Acid	-	-	5.3	-	Calcium Hydroxide		12.4 (sat)		
Carbonic Acid		3.8 (sat)			Ferrous Hydroxide		9.5 (sat)		
Citric Acid	-	2.6	2.1	-	Lime (CaO)		12.4 (sat)		
Formic Acid	-	-	2.3	-	Magnesia - Mg(OH) ₂		10.5 (sat)		
Hydrochloric Acid	3.0	2.0	1.1	0.1	Potassium Acetate	-	-	9.7	-
Hydrocyanic Acid	-	-	5.1	-	Potassium Bicarbonate	-	-	8.3	-
Hydrogen Sulfide	-	-	4.1	-	Potassium Carbonate	-	-	11.5	-
Lactic Acid	-	-	2.4	-	Potassium Cyanide	-	-	11.0	-
Malic Acid	-	-	2.2	-	Potassium Hydroxide	11.0	12.0	13.0	14.0
Orthophosphoric Acid	-	-	1.5	-	Sodium Acetate	-	-	8.9	-
Oxalic Acid	-	-	1.3	-	Sodium Benzoate	-	-	8.0	-
Succinic Acid	-	-	2.7	-	Sodium Bicarbonate	-	-	8.4	-
Salicylic Acid		2.4 (sat)			Sodium Carbonate	-	11.0	11.6	-
Sulfuric Acid	-	2.1	1.2	0.3	Sodium Hydroxide	11.1	12.0	13.0	14.0
Sulfurous Acid	-	-	1.5	-	Sodium Metasilicate	-	-	12.6	-
Tartaric Acid	-	-	2.0	-	Sodium Sesquicarbonate	-	-	10.1	-
Trichloroacetic Acid	-	-	1.2	-	Trisodium Phosphate	-	-	12.0	-

(sat) = saturated solution

Sources: [107, 190, 229, 253, 267]

Recipes for Dilute Acid/Base Solutions

By Volume: Volume (mL) of Concentrated Reagent per Liter of Solution

	Wt. %	Normal Solutions					Molar Solutions				
		0.05	0.1	0.5	1.0	5.0	0.05	0.1	0.5	1.0	5.0
Acetic Acid, glacial	99.5	2.9	5.7	28.7	57.5	287.4	2.9	5.7	28.7	57.5	287.4
	36	8.0	16.0	79.8	159.6	798.1	8.0	16.0	79.8	159.6	798.1
Butyric Acid	95	4.8	9.7	48.3	96.6	483.1	4.8	9.7	48.3	96.6	483.1
Formic Acid	90	2.1	4.2	21.0	41.9	209.6	2.1	4.2	21.0	41.9	209.6
Hydriodic Acid	57	6.6	13.2	66.0	132.0	660.0	6.6	13.2	66.0	132.0	660.0
	47	9.1	18.1	90.7	181.4	907.1	9.1	18.1	90.7	181.4	907.1
Hydrobromic Acid	48	5.5	11.1	55.4	110.9	554.5	5.5	11.1	55.4	110.9	554.5
	40	7.3	14.7	73.3	146.6	732.9	7.3	14.7	73.3	146.6	732.9
Hydrochloric Acid	37	4.3	8.6	42.8	85.7	428.4	4.3	8.6	42.8	85.7	428.4
	10	17.4	34.7	173.6	347.2	-	17.4	34.7	173.6	347.2	-
Hydrofluoric Acid	48	1.8	3.7	18.4	36.9	184.5	1.8	3.7	18.4	36.9	184.5
Hypophosphorous Acid	50	1.8	3.5	17.6	35.2	176.0	5.3	10.6	52.8	105.6	528.0
	70	3.2	6.3	31.7	63.4	316.9	3.2	6.3	31.7	63.4	316.9
Nitric Acid	67	3.4	6.7	33.6	67.2	335.8	3.4	6.7	33.6	67.2	335.8
	70	4.3	8.6	43.0	85.9	429.7	4.3	8.6	43.0	85.9	429.7
Perchloric Acid	60	5.4	10.9	54.4	108.7	543.6	5.4	10.9	54.4	108.7	543.6
Phosphoric Acid	85	1.1	2.2	11.2	22.5	112.4	3.4	6.7	33.7	67.4	337.1
Sulfuric Acid	96	1.4	2.8	13.9	27.8	138.8	2.8	5.6	27.8	55.5	277.7
Sulfurous Acid	6	33.5	67.1	335.3	670.6	-	67.1	134.1	670.6	-	-
Ammonia (Ammonium Hydroxide)	28	3.4	6.8	33.8	67.6	337.9	3.4	6.8	33.8	67.6	337.9
	50	3.7	7.4	36.9	73.8	369.1	3.7	7.4	36.9	73.8	369.1
Potassium Hydroxide	45	4.3	8.5	42.7	85.4	427.0	4.3	8.5	42.7	85.4	427.0
	10	25.7	51.5	257.4	514.8	-	25.7	51.5	257.4	514.8	-
Sodium Hydroxide	50	2.6	5.2	26.1	52.3	261.4	2.6	5.2	26.1	52.3	261.4
	10	18.0	36.0	180.2	360.4	-	18.0	36.0	180.2	360.4	-

The table above gives the volume (in mL) of the concentrated reagent that must be used to prepare 1 liter of aqueous solution of the concentration shown. Note the distinction between molarity and normality. A 1 M solution means 1 mole/L of the acid or base species, whereas a 1N solution is defined as 1 mole/L of dissociable H^+ or OH^- groups. Thus for multi-protic acids or bases, N and M are not equal. H_2SO_4 for example has two ionizable protons, and therefore a 1M solution is equal to a 2N solution.

The equations below can be used to calculate the reagent volume required for 1 liter of solution at various concentrations, where **mw** = species molecular weight, **M** or **N** equals the desired molarity or normality, weight percent (wt%) and density refer to the concentrated reagent and basicity is the number of removable protons (i.e. $HCl=1$, $H_2SO_4=2$, $H_3PO_4=3$, etc.):

For Molar Solutions

$$\text{Vol (ml)} = \frac{100 \times \text{mw} \times \text{M}}{\text{wt\%} \times \text{density}}$$

For Normal Solutions

$$\text{Vol (ml)} = \frac{100 \times \text{mw} \times \text{N}}{\text{wt\%} \times \text{basicity} \times \text{density}}$$

Recipes for Dilute Acid/Base Solutions

By Weight: Weight (g) of Concentrated Reagent per Kg of Solution

	Wt. %	Desired Wt% Solution									
		0.1	0.5	1.0	2.0	5.0	10.0	20.0	30.0	40.0	50.0
Acetic Acid, glacial	99.5	1.0	5.0	10.1	20.1	50.3	100.5	201.0	301.5	402.0	502.5
	36	2.8	13.9	27.8	55.6	138.9	277.8	555.6	833.3	-	-
Butyric Acid	95	1.1	5.3	10.5	21.1	52.6	105.3	210.5	315.8	421.1	526.3
Formic Acid	90	1.1	5.6	11.1	22.2	55.6	111.1	222.2	333.3	444.4	555.6
Hydriodic Acid	57	1.8	8.8	17.5	35.1	87.7	175.4	350.9	526.3	701.8	877.2
	47	2.1	10.6	21.3	42.6	106.4	212.8	425.5	638.3	851.1	-
Hydrobromic Acid	48	2.1	10.4	20.8	41.7	104.2	208.3	416.7	625.0	833.3	-
	40	2.5	12.5	25.0	50.0	125.0	250.0	500.0	750.0	-	-
Hydrochloric Acid	37	2.7	13.5	27.0	54.1	135.1	270.3	540.5	810.8	-	-
	10	10.0	50.0	100.0	200.0	500.0	-	-	-	-	-
Hydrofluoric Acid	48	2.1	10.4	20.8	41.7	104.2	208.3	416.7	625.0	833.3	-
Hypophosphorous Acid	50	2.0	10.0	20.0	40.0	100.0	200.0	400.0	600.0	800.0	-
	70	1.4	7.1	14.3	28.6	71.4	142.9	285.7	428.6	571.4	714.3
Nitric Acid	67	1.5	7.5	14.9	29.9	74.6	149.3	298.5	447.8	597.0	746.3
	70	1.4	7.1	14.3	28.6	71.4	142.9	285.7	428.6	571.4	714.3
Perchloric Acid	60	1.7	8.3	16.7	33.3	83.3	166.7	333.3	500.0	666.7	833.3
Phosphoric Acid	85	1.2	5.9	11.8	23.5	58.8	117.6	235.3	352.9	470.6	588.2
Sulfuric Acid	96	1.0	5.2	10.4	20.8	52.1	104.2	208.3	312.5	416.7	520.8
Sulfurous Acid	6	16.7	83.3	166.7	333.3	833.3	-	-	-	-	-
Ammonia (Ammonium Hydroxide)	28	3.6	17.9	35.7	71.4	178.6	357.1	714.3	-	-	-
	50	2.0	10.0	20.0	40.0	100.0	200.0	400.0	600.0	800.0	-
Potassium Hydroxide	45	2.2	11.1	22.2	44.4	111.1	222.2	444.4	666.7	888.9	-
	10	10.0	50.0	100.0	200.0	500.0	-	-	-	-	-
Sodium Hydroxide	50	2.0	10.0	20.0	40.0	100.0	200.0	400.0	600.0	800.0	-
	10	10.0	50.0	100.0	200.0	500.0	-	-	-	-	-

The table above gives the weight (in g) of the concentrated reagent that must be used to prepare 1 kg of aqueous solution of the concentration shown. In larger-scale operations, weight should be used instead of volume because it is intrinsically more accurate and not subject to change as temperature varies.

The equation below can also be used to prepare dilute solutions by weight. The equation calculates the weight, in grams, of the concentrated reagent required to prepare 1 kg of solution of the desired concentration.

$$\text{Weight (g)} = 1000 \times \frac{\text{desired concentration (wt\%)}}{\text{reagent concentration (wt\%)}}$$

In general, dilutions of any solutions can be easily calculated by using the simple equivalency principle taught in elementary chemistry:

$$\text{Starting Concentration} \times \text{Starting Volume} = \text{Final Concentration} \times \text{Final Volume}$$

$$\text{or} \quad C_o \times V_o = C_f \times V_f$$

pKa of Acids, Bases and Buffers

The tables on these pages list the room temperature pKa values of some common acids, bases, amino acids and buffer salts in water. The small numerals indicate the particular ionization step for multiprotic acids or multihydroxylated bases. In the case of bases, the value reported is the pKa of the conjugate acid. The pKa's of the amino acids listed correspond to the ionization of the carboxylic acid group, the basic amine group or another ionizable R-group in the molecule. Values in parenthesis are approximate because precise measurements cannot be made.

pKa of Common Acids in Water

Acid	pKa (RT)	Acid	pKa (RT)	Acid	pKa (RT)
Acetic	4.76	1,1-Cyclohexanediacetic	3.52 ¹	Maleic	2 ¹
Arsenic	2.25 ¹		6.94 ²		6.26 ²
	6.77 ²	1,1-Cyclopentanediacetic	3.82 ¹	Malic	3.4 ¹
	11.6 ³		6.70 ²		5.05 ²
Arsenious	9.23		3.07 ¹	Malonic	2.85 ¹
	4.10 ¹	Cyclopentanetetra-1,2,3,4-	4.48 ²		6.1 ²
Ascorbic	11.79 ²	carboxylic	5.57 ³	Mandelic	3.36
Barbituric	3.98		10.06 ⁴		3.53 ¹
	2.08 ¹	Dibenzoyltartaric acid	5.85 ¹	2-Methylpropane-1,2,3-	5.02 ²
	2.46 ²		7.50 ²	tricarboxylic	7.2 ³
	3.24 ³	2,2-Dimethylglutaric	4.31 ¹	Nitric	-1.64
Benzenehexacarboxylic	4.44 ⁴		5.51 ²	Nitrous	3.4
	5.5 ⁵	3,3-Dimethylglutaric	3.79 ¹	Octanoic	4.89
	6.59 ⁶		6.31 ²	Oxalic	1.19 ¹
	2.98 ¹	Dimethylmalonic	3.29 ¹		4.21 ²
Benzene-1,2,3-tricarboxylic	4.25 ²		5.98 ²	Perchloric	(-8)
	5.87 ³	2,2-Dimethylsuccinic	4.11 ¹	Periodic	1.64
	2.43 ¹		6.29 ²		2.12 ¹
Benzene-1,2,4,5-	3.13 ²		1.70 ¹	Phosphoric	7.21 ²
tetracarboxylic	4.44 ³	EDTA (Ethylenediamine	2.60 ²		12.32 ³
	5.61 ⁴	tetraacetic acid)	6.30 ³	Phosphorous	1.8 ¹
	3.16 ¹		10.60 ⁴		6.2 ²
Benzene-1,3,5-tricarboxylic	3.98 ²	Formic	3.75	Phthalic	2.9
	4.85 ³	Fumaric	3.03 ¹		3.1 ¹
	2.34 ¹		4.47 ²	o-Phthalic	5.27 ²
	2.95 ²	Glycerophosphoric	1.46 ¹	Picric	0.38
Benzenepentacarboxylic	3.94 ³		6.19 ²	Picolinic	5.4
	5.07 ⁴	Glycylglycine	3.06		5.03
	6.25 ⁵	Hexanoic	4.85	Pivalic (trimethylacetic)	3.67 ¹
Benzoic	4.20	Hippuric	3.64	Propane-1,2,3-tricarboxylic	4.84 ²
	2.30 ¹	Hydratropic	8.45		6.2 ³
Brucine tetrahydrate	7.95 ²	Hydrobromic	(-9)	Propionic	4.87
	3.36 ¹	Hydrocyanic	9.31		0.85 ¹
	4.38 ²	Hydrofluoric	3.45	Pyrophosphoric	1.96 ²
Butane-1,2,3,4-tetracarboxylic	5.45 ³		7.04 ¹		6.68 ³
	6.63 ⁴	Hydrogen sulfide	11.96 ²		9.39 ⁴
n-Butyric	4.82	Hydriodic	(-11)	Salicylic	2.98
Camphorsulfonic Acid	2.00		4.08 ¹		4.19 ¹
	6.37 ¹	3-Hydroxybenzoic	9.92 ²	Succinic	5.57 ²
Carbonic (H ₂ CO ₃)	10.25 ²		8.69		(-3) ¹
Chloroacetic	2.87	Hypobromous	7.53	Sulfuric	1.92 ²
	0.74 ¹	Hypochlorous	10.00		1.82 ¹
Chromic	6.49 ²	Hypoiodous	0.77	Sulfurous	6.91 ²
	3.06 ¹	Iodic	3.84 ¹		3.02 ¹
	4.74 ²	Itaconic	5.55 ²	Tartaric	4.54 ²
Citric	5.40 ³	Lactic	3.86	Trifluoroacetic	0.23

1 = first ionization step, 2 = 2nd ionization step, etc.

Sources for pKa Tables: [70, 149, 154, 190, 229, 232, 255, 267]

pKa of Common Bases in Water

Base	pKa (RT)	Base	pKa (RT)	Base	pKa (RT)
Ammonia	9.25	Ethylamine	10.80	Piperazine	9.83 ¹
Aniline	4.63	Ethylene diamine	10.08 ¹		5.56 ²
Benzylamine	9.33	Glycinamide	6.99 ²	Purine	2.39
	9.24 ¹		8.15	Pyrazine	0.65 ¹
Boric acid	12.74 ²	Hexamethylene diamine	11.10 ¹		5.21 ²
	13.80 ³		10.01 ²	Pyridine	5.23
n-Butylamine	10.78	Hydroxylamine	6.03	Pyrimidine	0.65
Cyclohexylamine	10.66	Imidazole	6.95	Pyrrolidine	11.27
Diethanolamine	8.90	Isoquinoline	5.38	Quinoline	4.87
Diethylamine	10.98	Methylamine	10.64	Taurine	9.10
Diisopropylamine	11.13	N-Methylalanine	4.85	m-Toluidine	4.73
Diisopropylethylamine	10.40	a-Methylbenzylamine	9.50	o-Toluidine	4.45
Dimethylamine	10.72	N-Methylpiperidine	10.08	p-Toluidine	5.08
4-Dimethylaminopyridine	9.70	N-Methylpyrrolidone	10.32	Triethanolamine	7.75
Diphenylamine	0.79	Morpholine	8.33	Triethylamine	10.75
Ephedrine	9.70	N-Glycyl-glycine	8.40	Trimethylamine	9.74
Ethanolamine	9.44	Piperidine	11.12	Urea	0.10

pKa of Common Amino Acids in Water

Amino acid	pKa (a) at RT	pKa (b) at RT	pKa (R) at RT	Amino acid	pKa (a) at RT	pKa (b) at RT	pKa (R) at RT
Alanine	2.35	9.69	-	Lysine	2.18	8.95	10.53
Arginine	2.17	9.04	12.48	Methionine	2.28	9.21	-
Asparagine	2.02	8.8	-	Phenylalanine	1.83	9.13	-
Aspartic Acid	2.09	9.82	3.86	Proline	1.99	10.6	-
Cysteine	1.71	10.78	8.33	Serine	2.21	9.15	-
Glutamine	2.17	9.13	-	Taurine	1.5	8.74	-
Glutamic Acid	2.19	9.67	4.25	Threonine	2.63	10.43	-
Glycine	2.34	9.6	-	Tryptophan	2.38	9.39	-
Histidine	1.82	9.17	6	Tyrosine	2.2	9.11	10.07
Leucine	2.36	9.6	-	Valine	2.32	9.62	-

pKa of Common Organic Buffer Salts in Water

Buffer Salt	pKa (RT)	Buffer Salt	pKa (RT)
"ACES" N-(2-Acetamido)-2-aminoethanesulfonic acid	6.9 ²	"HEPES" N-2-Hydroxyethyl piperazine-N'-2-ethanesulfonic acid	7.55 ²
"ADA" N-(2-Acetamido)imino-diacetic acid	6.6 ³	"MES" 2-(N-Morpholino)-ethane sulfonic acid	6.15 ¹
"BES" N,N-Bis(2-hydroxyethyl)-2-aminoethane-sulfonic acid	7.15 ²	"MONO-TRIS" 2-Hydroxyethylimino-tris(hydroxymethyl)methane	7.83
"BICINE" N,N-Bis(2-hydroxyethyl)glycine	8.35	"MOPS" 2-(N-Morpholino)propane-sulfonic acid	7.2 ²
"BIS-TRIS" Bis(2-hydroxyethyl)imino-tris(hydroxymethyl)-methane	6.46		3.0 ³
"CAPS" 3-Cyclohexylamino-1-propanesulfonic acid	10.4 ²	"PIPES" 1,4-Piperazinebis-(ethanesulfonic acid)	6.8 ⁴
"CHOLAMINE" 2-(Aminoethyl)trimethylammonium chloride	7.1		
	4.3 ¹	"TAPS" N-Tris(hydroxymethyl)methyl-2-amino-propane sulfonic acid	8.4 ²
"EMTA" 3,6-Endomethylene-1,2,3,6-tetrahydro-phthalic acid	7.0 ²	"TES" N-Tris(hydroxymethyl)methyl-2-aminoethane sulfonic acid	7.5 ²
		"TRICINE" N-Tris(hydroxymethyl)methylglycine	8.15
"EPPS" 4-(2-Hydroxyethyl)-1-piperazinepropane sulfonic acid	8.0	"TRIS" Tris(hydroxymethyl)aminomethane	8.1

Properties and Preparation of Buffer Solutions

Buffers are solutions of acids, bases or acid-base salts that can maintain relatively constant pH in spite of changes in acid or base concentration. Compounds used for this purpose exhibit a characteristic pH value (called the pKa) at which they resist changes in pH, because at this point the concentrations of their protonized and de-protonized forms are equal. This represents the flattest part of the pH titration curve, the point at which the pH changes little upon the addition of acid or base.

In simple terms, when an acid is dissolved in water, it partly dissociates into its de-protonized form and a proton. The protonized and de-protonized forms are often called a conjugate acid-base pair. The degree of dissociation is quantified by the pH value, since the pH is related to the concentration of protons (H^+) in solution ($pH = -\log [H^+]$). A more complete discussion of pH and its derivation can be found in most elementary chemistry texts.

Aqueous buffers can be prepared from any ionizable salts of acids or bases such as those listed on the previous pages, but certain ones are most commonly used (see page 8-9). In preparing a buffer, select a compound whose pKa is within 0.5 pH units of the desired pH (at 1 pH unit away from the pKa, the buffering power is only about 1/3 of that at the pKa). The pH of the buffer solution is then adjusted up or down as necessary, usually using HCl, NaOH or KOH. For multi-protic acids, the pH of the unadjusted solution will tend to equal the average of its multiple pKa values, but for mono-protic acids the pH will depend on concentration and other factors. The concentration of the solution, and thus its ionic strength, is somewhat arbitrary, but it is economical to use the lowest concentration that will fit the purpose.

It is sometimes possible to prepare a buffer directly by mixing the protonated and de-protonated forms of the salt in the appropriate ratio. The following useful relationship (called the Henderson-Hasselbalch equation), which holds true for any conjugate acid-base pair, relates the pH of a buffer solution to its composition and pKa:

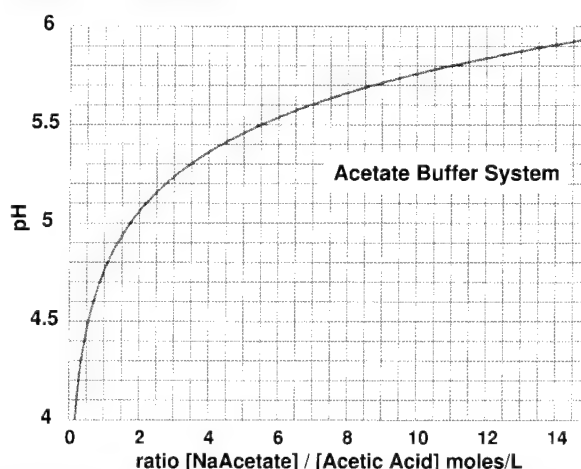
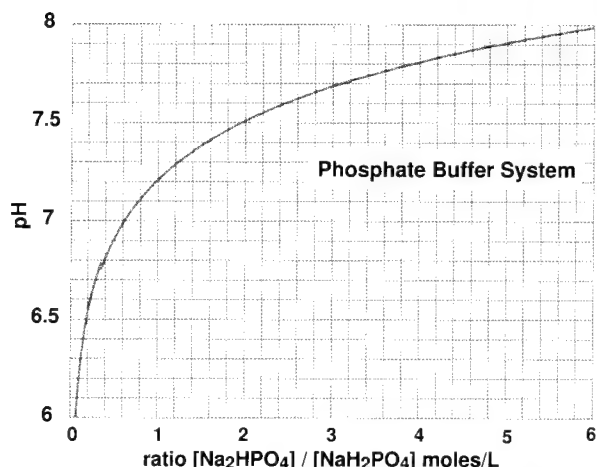
$$pH = pKa + \log \frac{[\text{de-protonated form}]}{[\text{protonated form}]} \quad \text{example: } \frac{[HPO_4^{2-}]}{[H_2PO_4^-]}$$

For example, in the case of the common pH 7 phosphate buffer system, the pH is determined by the concentration of the protonated ($H_2PO_4^-$) and the de-protonated (HPO_4^{2-}) forms associated with the 2nd-step pKa of 7.2. Thus a buffer of the desired pH can be prepared by mixing monobasic Na_2HPO_4 and dibasic NaH_2PO_4 in water in the appropriate ratio. The charts below show typical compositions for two common buffer systems, phosphate and acetate.

Stronger buffers can often be obtained by selecting two compounds, one with a pKa slightly above and one slightly below the desired pH and mixing them in an appropriate ratio. Always check the pH of a buffer with a pH meter, calibrated with a standardized buffer at the temperature at which the solution will be used. Also remember that the pH of the buffer may change upon dilution or in the presence of organic solvents or other ions.

Phosphate and Acetate Buffer Compositions

(actual ratio may vary slightly depending on concentration)



Some Useful Buffer Systems

Buffer	Useful pH Range	Buffer	Useful pH Range
Glycine and HCl	1.0 - 3.7	"HEPES" Buffer and NaOH	6.9 - 8.3
Citrate and HCl	1.3 - 4.7	Triethanolamine and HCl	6.9 - 8.5
Formate and HCl	2.8 - 4.6	"TRIS" Buffer and HCl	7.2 - 9.0
Succinic Acid and Na ₂ B ₄ O ₇	3.0 - 5.8	"Tricine" Buffer and HCl	7.2 - 9.0
Acetate and Acetic Acid	3.7 - 5.6	"Bicine" Buffer and HCl	7.4 - 9.2
Succinate and Succinic Acid	4.8 - 6.3	Na ₂ B ₄ O ₇ and HCl	7.6 - 8.9
"MES" Buffer and NaOH	5.2 - 7.1	Glycine and NaOH	8.2 - 10.1
KH ₂ PO ₄ and Na ₂ B ₄ O ₇	5.8 - 9.2	Ethanolamine and HCl	8.6 - 10.4
KH ₂ PO ₄ and K ₂ HPO ₄	6.1 - 7.5	Na ₂ CO ₃ and NaHCO ₃	9.2 - 11.1
"TES" Buffer and NaOH	6.8 - 8.2	Na ₂ HPO ₄ and NaOH	11.0 - 12.0

Sources: [70, 154, 190, 229, 232, 267]

Some NBS Standardized Buffers

Buffer	pH at 20°C	Approx. Change in pH / 10°C increase	Approx. Change in pH upon 2:1 dilution
0.05M Potassium Tetraoxalate	1.68	+0.005	0.186
sat. (25°C) Potassium Hydrogen Tartrate	3.55	-0.008	0.049
0.05M Potassium Dihydrogen Citrate	3.79	-0.036	0.024
0.05M Potassium Hydrogen Phthalate	4.00	+0.016	0.052
0.025 M KH ₂ PO ₄ , 0.025M Na ₂ HPO ₄	6.88	-0.051	0.08
0.0087 M KH ₂ PO ₄ , 0.0304M Na ₂ HPO ₄	7.43	-0.052	0.07
0.01M Na ₂ B ₄ O ₇	9.23	-0.119	0.01
0.025M NaHCO ₃ , 0.025M Na ₂ CO ₃	10.06	-0.127	0.079
sat. (25°C) Ca(OH) ₂	12.63	-0.398	-0.028

Sources: [70, 229, 267]

pH Range of Some Acid-Base Color Indicators

Indicator	pH range / color change	Indicator	pH range / color change
Methyl Violet	0.0-1.6 yell to blue	Azolitmin	5.0-8.0 red to blue
Methyl Green	0.2-1.8 yell to blue	Bromocresol Purple	5.2-6.8 yell to purp
Quinaldine Red	1.0-2.2 col to red	Bromothymol Blue	6.0-7.6 yell to blue
Mentanil Yellow	1.2-2.4 red to yell	Neutral Red	6.8-8.0 red to amb
4-Phenylazodiphenylamine	1.2-2.6 red to yell	Cresol Red	7.0-8.8 yell to red
Orange IV	1.4-2.8 red to yell	a-Naphtholphthalein	7.3-8.7 rose to grn
Erythrosine, disodium salt	2.2-3.6 or to red	Tropeolin 000	7.6-8.9 yell to rose-red
Methyl Yellow	2.9-4.0 red to yell	Phenolphthalein	8.2-10.0 col to pink
Bromophenol Blue	3.0-4.6 yell to blue	o-Cresolphthalein	8.2-9.8 col to red
Methyl Orange	3.2-4.4 red to yell	Thymolphthalein	9.4-10.6 col to blue
Ethyl Orange	3.4-4.8 red to yell	Nile blue	10.1-11.1 blue to red
a-Naphthyl red	3.7-5.0 red to yell	Nitramine	10.8-13.0 col to brown
Bromocresol Green	3.8-5.4 yell to blue	Alizarin	11.0-12.4 red to purp
Ethyl Red	4.0-5.8 col to red	2,4,6-Trinitrotoluene	11.5-13.0 col to or
Alizarin Red S	4.6-6.0 yell to red	Trinitrobenzoic acid (ind. salt)	12.0-13.4 col to or-red
Methyl Red	4.8-6.0 red to yell	1,3,5- Trinitrobenzene	12.0-14.0 col to or

col = colorless

Sources: [70, 154, 229, 267]

Preparation of the above indicators varies, but most are used at concentrations between 0.1 and 1% in water, ethanol or very dilute NaOH. The Merck Index [267] contains more detailed information as well as a comprehensive list of mixed indicators that often give much sharper endpoints.

Aqueous Solubility

The tables and charts that follow list the aqueous solubility of a number of representative compounds and some general rules that may prove useful. Solubility here means the saturation concentration, at which the solute is in equilibrium with excess undissolved solids in the mixture, and no more solids can be dissolved without heating. Under certain conditions, solutions of higher concentration can be prepared (supersaturated solutions) but they are unstable and will revert back to their saturated state as a result of mechanical shock, cooling or simply standing for a time.

The solubilities values given here should be considered approximate because of the considerable variation found in the literature reports, differences in grades and materials provided by various manufacturers and because various estimations were required when converting the available data into consistent units. Additional water-solubility data for certain solvents may be found in Chapter 6. The data provided here are by no means exhaustive, but will serve to indicate general trends. More complete lists can be found in references [154] and [267]. The effect of temperature on solubility is described in a graph of some representative compounds on page 8-13, again to present trends and examples of the various types of solubility curves that may be encountered in practice.

Measuring Solubility – This section can serve as a general guide, but since it is rare that pure solvents and solutes are found in practice, for critical applications solubility should be measured under actual process conditions. This is generally accomplished by preparing saturated solutions of the test compound at various temperatures and assaying the liquors (supernatant or filtrate) or weighing the undissolved solids. It is important that sufficient agitation be provided and time allowed for the mixture to reach equilibrium before sampling. In some cases, a time span of several days may be required to establish thermodynamic equilibrium in which case maintaining constant temperature and preventing evaporation become important concerns. It is useful to plot the data on a semilog plot vs. inverse temperature (see page 2-22). For solutions reasonably close to ideal, such a plot will result in a straight line, which simplifies interpolation.

General Water Solubility Rules for Inorganic Compounds

Acetates	All soluble. AgCH_3CO_2 moderately.
Ammonium	All salts soluble except $(\text{NH}_4)_2\text{PtCl}_6$ and $(\text{NH}_4)_2\text{NaCl}(\text{NO}_2)_6$.
Carbonates	All insoluble except Na, K and NH_4 carbonates.
Chlorides	All soluble except AgCl , Hg_2Cl_2 . PbCl_2 moderately in cold water, soluble in hot.
Chromates	All insoluble except Na, K and NH_4 chromates and MgCrO_4 .
Hydroxides	All insoluble except Li, Na, K, Ce, Ru, NH_4 . $\text{Ba}(\text{OH})_2$, $\text{Ca}(\text{OH})_2$ and $\text{Sr}(\text{OH})_2$ slightly.
Nitrates	All soluble.
Phosphates	All insoluble except Na, K and NH_4 phosphates.
Potassium	All salts soluble except $\text{K}_2\text{NaCo}(\text{NO}_2)_6$ and K_2PtCl_6 .
Silicates	All insoluble except Na, K and NH_4 silicates.
Silver	All salts insoluble except AgNO_3 and AgClO_4 . $\text{AgC}_2\text{H}_3\text{O}_2$ and Ag_2SO_4 moderately.
Sodium	All salts soluble except $\text{Na}_4\text{Sb}_2\text{O}_7$.
Sulfates	All soluble except BaSO_4 and PbSO_4 . Ag, Hg(I). CaSO_4 slightly. Hydrogen sulfates generally more sol. than sulfates.
Sulfides	All insoluble except Li, Na, K, NH_4 , Mg, Ca, Ba. Al and Cr hydrolyze and precipitate as hydroxides.

Sources: [70, 110, 232]

United States Pharmacopeia Definitions of Solubility

Term	Solvent Parts Required per 1 Part of Solute	Solubility (mg/mL)
Very Soluble	< 1	> 1000
Freely Soluble	1 - 10	100 - 1000
Soluble	10 - 30	33 - 100
Sparingly Soluble	30 - 100	10 - 30
Slightly Soluble	100 - 1000	1 - 10
Very Slightly Soluble	1000 - 10,000	0.1 - 1.0
Practically Insoluble or Insoluble	> 10,000	< 0.1

Source: [248]

Aqueous Solubility of Selected Inorganic Compounds

Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%
Alum. Amm. Sulfate ·24H ₂ O	12	Cobalt Chlorate	65	Nickel Nitrate ·6H ₂ O	45
Aluminum Chloride ·6H ₂ O	55	Cobalt Nitrate	51	Nickel Perchlorate	65
Aluminum Fluoride ·5H ₂ O	0.5	Cobalt Perchlorate	70	Nickel Perchlorate ·9H ₂ O	55
Aluminum Potass. Sulfate	6	Cu. Amm. Chloride ·2H ₂ O	28	Nickel Sulfate ·6H ₂ O	45
Aluminum Sulfate ·18H ₂ O	48	Cupric Ammonium Sulfate	15	Phosphomolyb. A. ·48H ₂ O	70
Ammonium Arsenate	32	Cupric Bromide	54	Phosphotungstic A. ·25H ₂ O	68
Ammonium Bromide	42	Cupric Chlorate	63	Potassium Acetate	65
Ammonium Carbonate	20	Cupric Nitrate ·6H ₂ O	56	Potassium Bicarbonate	25
Ammonium Chloride	27	Cupric Selenate	15	Potassium Bitartrate	0.6
Ammonium Dichromate	27	Cupric Sulfate ·5H ₂ O	18	Potassium Bromate	7
Ammonium Iodide	64	Ferric Ammonium Citrate	65	Potassium Bromide	38
Amm. Molybdate ·4H ₂ O	30	Ferr. Amm. Oxalate ·3H ₂ O	50	Potass. Carbonate ·1½H ₂ O	50
Ammonium Nitrate	902	Ferric Ammonium Sulfate	20	Potassium Chlorate	7
Ammonium Oxalate ·H ₂ O	5	Ferric Chloride	72	Potassium Chloride	25
Ammonium Perchlorate	20	Ferric Nitrate	45	Potassium Chromate	37
Ammonium Periodate	27	Ferr. Perchlorate ·10H ₂ O	75	Potassium Citrate	58
Ammonium Persulfate	42	Ferrous Sulfate ·7H ₂ O	40	Potassium Dichromate	12
Amm. Phosphate, Dibasic	58	Lead Acetate	35	Potassium Ferricyanide	32
Amm. Phosphate, Monobas.	27	Lead Bromide	1	Potassium Ferrocyanide	22
Ammonium Silicofluoride	16	Lead Chlorate	62	Potassium Fluoride ·2H ₂ O	50
Ammonium Sulfate	43	Lead Chloride	1	Potassium Formate	78
Ammonium Sulfite ·H ₂ O	39	Lead Iodide	0.1	Potassium Hydroxide	55
Ammonium Thiocyanate	61	Lead Nitrate	35	Potassium Iodate	8
Barium Bromide	51	Lithium Bromate	62	Potassium Iodide	58
Barium Chlorate	28	Lithium Carbonate	2	Potass. Meta-Antimonate	3
Barium Chloride	26	Lithium Chloride ·H ₂ O	42	Potassium Nitrate	25
Barium Iodide ·7.5H ₂ O	67	Lithium Citrate	30	Potassium Nitrite	74
Barium Nitrate	9	Lithium Dichromate ·H ₂ O	53	Potassium Oxalate ·H ₂ O	26
Barium Nitrite	42	Lithium Fluoride	0.3	Potassium Perchlorate	2
Barium Perchlorate	75	Lithium Formate	30	Potassium Periodate	0.7
Beryllium Sulfate ·4H ₂ O	27	Lithium Iodate	45	Potassium Permanganate	6
Cadmium Bromide ·4H ₂ O	51	Lithium Nitrate	50	Potassium Stannate	45
Cadmium Chlorate ·12H ₂ O	78	Lithium Perchlorate ·3H ₂ O	35	Potassium Sulfate	9
Cadmium Chloride ·2½H ₂ O	53	Lithium Sulfate ·H ₂ O	25	Rubidium Bromate	3
Cadmium Iodide	45	Magn. Bromide ·6H ₂ O	52	Rubidium Bromide	49
Cadmium Sulfate ·8H ₂ O	42	Magnesium Chlorate	58	Rubidium Chloride	45
Calcium Bromide	59	Magn. Chloride ·6H ₂ O	60	Rubidium Iodate	3
Calcium Chlorate ·2H ₂ O	65	Magn. Chromate ·7H ₂ O	45	Rubidium Iodide	60
Calcium Chloride ·6H ₂ O	44	Magn. Dichromate ·5H ₂ O	78	Rubidium Nitrate	37
Calcium Chromate ·2H ₂ O	15	Magnesium Iodate ·4H ₂ O	6	Rubidium Perchlorate	2
Calcium Ferrocyanide	35	Magnesium Iodide ·8H ₂ O	60	Rubidium Periodate	1
Calcium Iodide	68	Magnesium Molybdate	15	Rubidium Sulfate	30
Calcium Nitrite ·4H ₂ O	46	Magnesium Nitrate ·6H ₂ O	40	Silver Bromate	0.2
Calcium Sulfate ·2H ₂ O	0.2	Magn. Perchlorate ·6H ₂ O	48	Silver Fluoride ·2H ₂ O	66
Carbon Disulfide	0.2	Magnesium Selenate	35	Silver Nitrate	70
Cerium Nitrate ·6H ₂ O	60	Magnesium Sulfate ·7H ₂ O	53	Silver Perchlorate ·H ₂ O	84
Cesium Bromide	53	Manganese Chloride	41	Sodium Acetate	30
Cesium Chloride	64	Manganese Nitrate ·6H ₂ O	60	Sodium Ammon. Sulfate	30
Cesium Iodide	47	Manganese Silicofluoride	40	Sodium Arsenate ·12H ₂ O	25
Cesium Nitrate	20	Manganese Sulfate	35	Sodium Bicarbonate	9
Cesium Perchlorate	2	Mercuric Bromide	0.5	Sodium Bisulfate	55
Cesium Periodate	3	Mercury Bichloride	6	Sodium Bromide ·2H ₂ O	45
Cesium Sulfate	63	β-Naphthalenesulf. Acid	50	Sodium Carbonate ·10H ₂ O	21
Chloral Hydrate ·H ₂ O	77	Nickel Amm. Sulfate ·6H ₂ O	8	Sodium Chlorate	50
Chromic Oxide	63	Nickel Chlorate	58	Sodium Chloride	26
Chrom. K Sulfate ·24H ₂ O	19	Nickel Chlorate ·6H ₂ O	66	Sodium Chromate	40

Aqueous Solubility of Selected Inorganic Compounds (continued)

Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%
Sodium Dichromate	65	Sod. Pyrophosphate ·6H ₂ O	12	Strontium Nitrite	40
Sodium Ferrocyanide	16	Sodium Salicylate	50	Strontium Perchlorate	74
Sodium Fluoride	3	Sodium Selenate	30	Thallium Chloride	0.4
Sodium Formate	45	Sodium Silicofluoride	0.8	Thallium Nitrate	10
Sodium Hydroxide	50	Sodium Sulfate	21	Thallium Nitrite	30
Sodium Hypophosphite	55	Sodium Sulfate ·10H ₂ O	26	Thallium Perchlorate	13
Sodium Iodate ·H ₂ O	8	Sodium Sulfide ·9H ₂ O	50	Thallium Sulfate	5
Sodium Iodide	64	Sodium Sulfite, Anh.	22	Trichloroacetic Acid	90
Sodium Molybdate	40	Sodium Thiocyanate	61	Uranyl Chloride	77
Sodium Nitrate	45	Sodium Thiosulfate ·5H ₂ O	65	Zinc Acetate	24
Sodium Nitrite	46	Sodium Tungstate ·10H ₂ O	44	Zinc Chlorate	65
Sodium Oxalate	3	Stannous Chloride	75	Zinc Chloride	65
Sodium Perchlorate	65	Strontium Chlorate	65	Zinc Iodide	82
Sodium Periodate ·3H ₂ O	12	Strontium Chloride ·6H ₂ O	35	Zinc Selenate	37
Sodium Phosphate Dibasic	4	Strontium Iodide ·6H ₂ O	64	Zinc Silicofluoride ·6H ₂ O	33
Sodium Phosphate Tribasic	10	Strontium Nitrate	44	Zinc Sulfate ·7H ₂ O	35

Sources: [70, 154, 194, 230, 232, 255, 259, 267]

Aqueous Solubility of Selected Organic Compounds

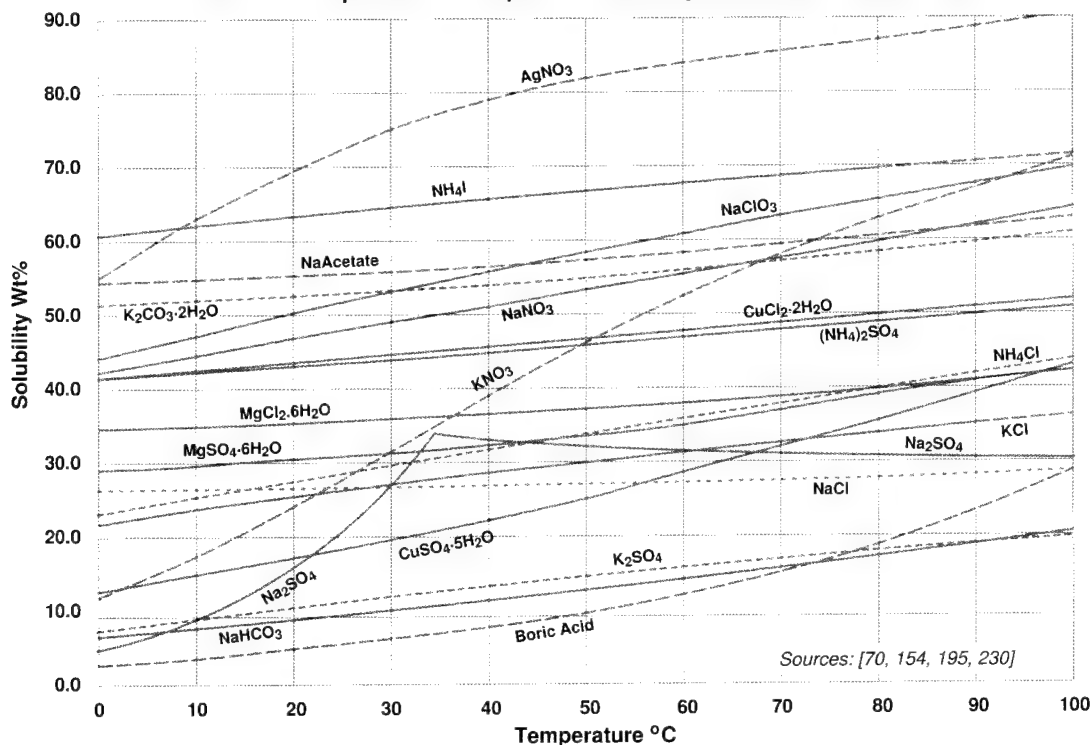
Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%
Acetamide	40.8	Carbon Disulfide	0.21	Dipropyl Ether	1.49
Acetophenone	0.55	Carbon Monoxide	0.003	Dipropylamine	2.5
Acrolein	20.8	Chlorobenzene	0.05	Ethyl Acetate	7.7
Alanine	13.9	1-Chloropropane	0.27	Ethyl Acrylate	1.5
o-Aminobenzoic Acid	0.5	Citric Acid ·H ₂ O	65.4	Ethyl Benzoate	0.083
DL-α-Aminoisobutyric Acid	13.0	Cresol, o-	3.08	Ethyl Formate	11.8
DL-α-Amino-n-Butyric Acid	17.3	Cycloheptane	0.003	Ethylbenzene	0.02
Ammonium Benzoate	18.0	Cyclohexane	0.006	Ethylene	0.0134
Ammonium Citrate, Dibasic	48.7	Cyclohexanol	4.3	1-Ethylhexylamine	0.25
Ammonium Salicylate	47.2	Cyclohexanone	2.3	Furan	1.0
Aniline	3.8	Cyclopentadiene	0.07	Furfural	8.3
Aniline Hydrochloride	44.4	Cyclopentane	0.016	Gallic Acid ·H ₂ O	1.1
Aniline Sulfate	5.9	Cyclopentene	0.054	D-Glutamic Acid	0.9
Anisole	0.19	Dextrose ·H ₂ O	47.7	Glycine	20.0
L-Asparagine	2.4	o-Dibromobenzene	0.007	Heptane	0.005
Benzaldehyde	0.3	Dibutyl Ether	0.03	1-Heptanol	0.17
Benzamide	1.3	Dibutylamine	0.47	Hexane	0.001
Benzene	0.18	o-Dichlorobenzene	0.014	Hexanoic Acid	0.96
Benzoic Acid	0.4	1,1-Dichloroethane	0.51	1-Hexanol	0.6
Benzyl Alcohol	0.03	1,2-Dichloroethane	0.81	2-Hexanol	1.4
Biphenyl	0.001	1,1-Dichloroethylene	0.04	Hexyl Acetate	0.02
Boric Acid	5.0	Dichloromethane	1.3	Hydroquinone	6.6
1,3-Butadiene	0.074	Diethanolamine	95.4	Indole	0.187
1-Butanol	7.5	1,1-Diethoxyethane	4.2	Isobutanol	8.7
2-Butanol	19.8	Diisobutyl Ketone	0.05	Isobutyl Acetate	0.6
2-Butanone	25.9	Diisopropyl Ether	1.2	Isobutyl Formate	1.0
Butyl Acetate	0.5	Dimethoxymethane	33	Isopropyl Acetate	2.9
1-Butyne	0.29	Dimethyl Ether	35.3	Lactose ·H ₂ O	15.9
Calcium Lactate ·5H ₂ O	4.9	Dimethyl Sulfide	2.0	DL-Leucine	1.0
Camphoric Acid	0.8	Dimethyl Sulfoxide	M	L-Leucine	2.2
Carbon Dioxide	0.15	Diphenyl Ether	0.002	d-Limonene	0.001

Aqueous Solubility of Selected Organic Compounds (continued)

Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%	Compound	Approx. sol. at Room T. Wt%
Lithium Benzoate	27.1	3-Pentanol	5.6	Succinimide ·H ₂ O	28.9
Lithium Salicylate	50.7	1-Pentanol	4.3	Sucrose	66.7
Mercuric Acetate	33.0	2-Pentanone	5.5	Tartaric Acid	58.8
Methacrylic Acid	8.9	Phenol	6.0	1,1,1,2-Tetrachloroethane	0.11
Methane	0.003	Phenyl Salicylate	0.0	Tetrachloroethylene	0.02
Methyl t-Butyl Ether	4.3	Phenyl Thiourea	0.2	Tetraethyl Amm. Iodide	32
Methyl Acetate	24.5	3-Phenylalanine	2.9	Tetramethyl Amm. Iodide	5.4
Methyl Acrylate	4.94	m-Phenylenediamine	21.7	Triacetin	5.8
Methyl Benzoate	0.21	p-Phenylenediamine	3.6	Tribromofluoromethane	0.04
Methyl Cyclohexane	0.002	Potassium Acetate	69.9	Trichloroacetic Acid	93.6
Methyl Formate	33.0	Pot. Sod. Tartrate ·4H ₂ O	43.0	1,2,3-Trichlorobenzene	0.003
Methyl Salicylate	0.74	Propanal	30.6	1,1,1-Trichloroethane	0.13
4-Methyl-2-pentanone	1.9	Propane	0.007	1,1,2-Trichloroethane	0.44
Methylacrylonitrile	2.57	Propyl Acetate	2.3	Trichloroethylene	0.11
Methylpropanoic Acid	22.8	Pyrrole	4.5	Trichloromethane	0.8
M-Hydroxybenzoic Acid	1.0	Quinine Salicylate	0.1	1,2,3-Trichloropropane	0.19
Monochloroacetic Acid	73.9	Quinoline	0.66	Triethylamine	miscible
Napthalene	0.002	Resorcinol	53.0	Urea	49.7
Nitrobenzene	0.19	Silver Acetate	1.1	Urethan	65.5
Nitroethane	4.7	Sodium Benzenesulfonate	16.4	Valine, D-	8.0
Nitromethane	miscible	Sodium Benzoate	35.6	Valine, DL-	6.5
1-Nonanol	0.014	Sodium Citrate ·5H ₂ O	49.0	Vinyl Acetate	2.0
DL-Norleucine	1.1	Sodium Phenolsulfonate	16.3	2,4-Xylenol	0.78
1-Octanol	0.6	Sodium Salicylate	52.8	Zinc Benzenesulfonate	30.6
Oxalic Acid ·2H ₂ O	9.9	Strontium Salicylate	4.6	Zn Phenolsulfonate ·8H ₂ O	39.8
Pentanoic Acid	2.4	Succinic Acid	7.6	Zinc Valerate	1.3

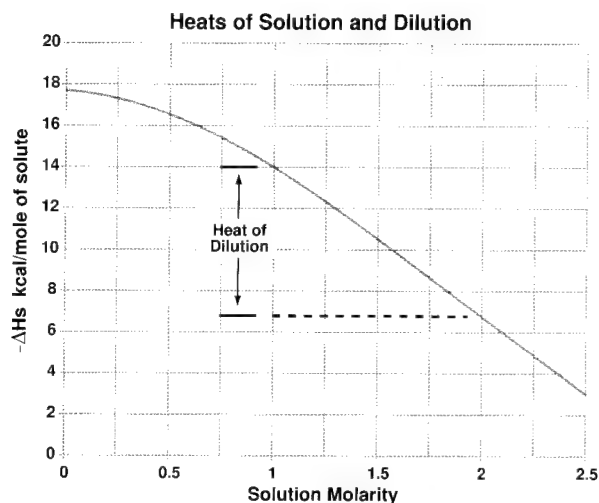
Sources: [70, 154, 194, 230, 232, 255, 259, 267]

Effect of Temperature on Aqueous Solubility of Selected Compounds



Heat of Solution

When a substance is dissolved in a solvent, when two solvents are mixed, or when a concentrated solution is diluted, there is an evolution or absorption of heat. This is called the heat (or enthalpy) of solution, ΔH_s . It can be an important consideration when making or diluting solutions of some substances, such as NaOH, in water or other polar solvents at large scale, where substantial amounts heat may be evolved and significant temperature increases may be observed.



In this example, 14 kcal/mole of heat is evolved in making a 1M solution. 7.2 kcal/mole (14 minus 6.8) is evolved when diluting a 2M solution to a 1M solution.

When an ideal solution is formed, there is no enthalpy change. However, real solutions are far from ideal, and various processes are at work when a solute dissolves. Generally speaking, energy must be absorbed to separate the ions in a crystal lattice. On the other hand, when there is a strong attraction between the solute ions and the solvent (solvation), energy is usually released. Ionization effects also play a role, and the net effect depends on which process dominates. Enthalpy change varies for different forms of salts, especially for different hydrates. Often, hydrated salts or those that form no hydrates dissolve endothermically; anhydrous salts that can undergo hydration dissolve exothermically.

It is important to note that the change in enthalpy per mole of solute is not a constant. Rather, it is a function of the final solution concentration. In most cases, the greatest energy change occurs at "infinite" dilution. As the concentration increases, less energy per mole of solute is evolved or absorbed. This is illustrated in the figure at left. The literature

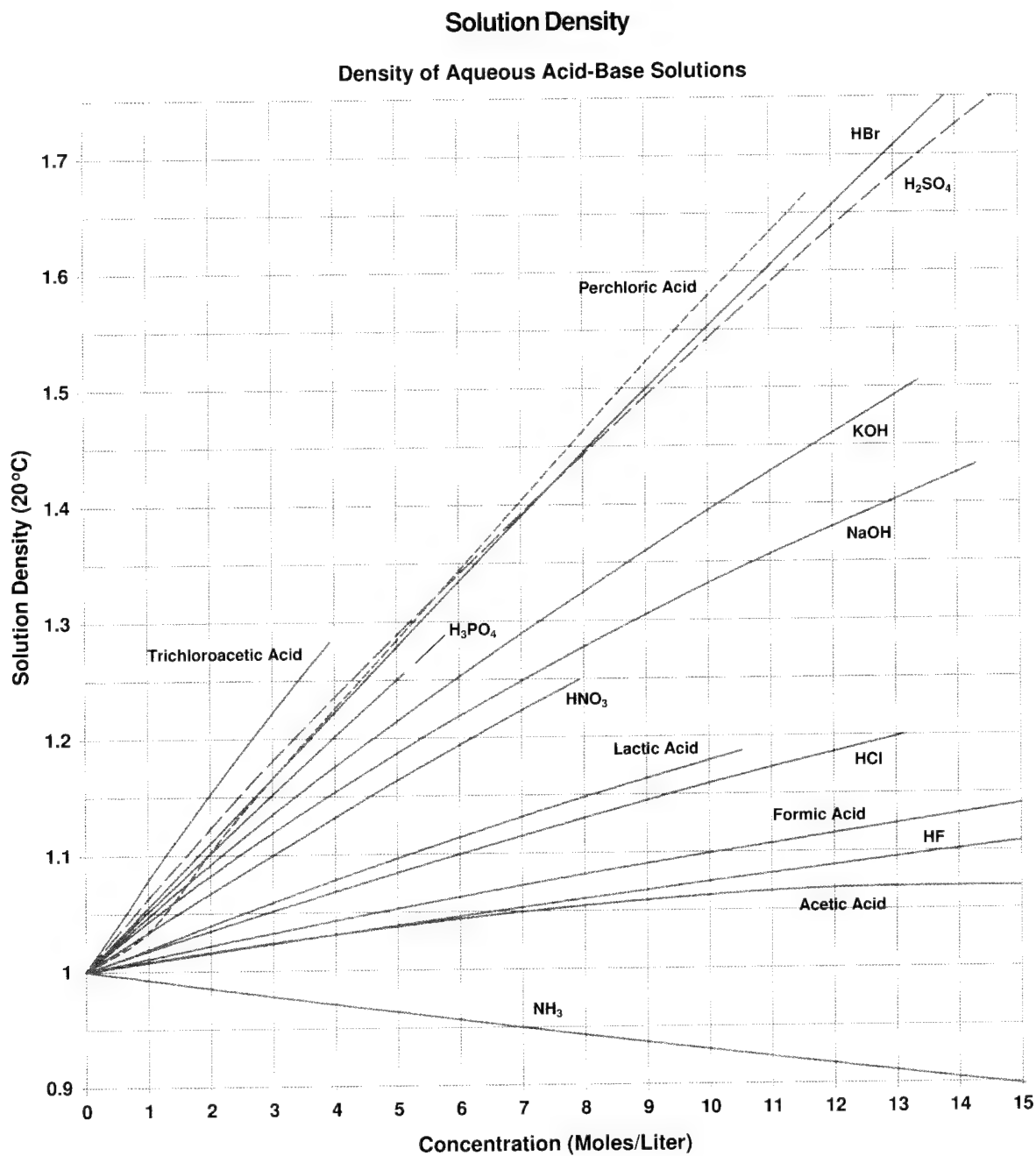
often reports a single value for ΔH_s , measured at a specific concentration or at infinite dilution. The relationship is complex, with no simple way to calculate ΔH_s exactly at a specific concentration without deriving it from elemental heats of formation in that particular solvent at that particular concentration. Rather, assume that the literature value represents the worst-case scenario, or, for critical applications, ΔH_s may be measured directly by calorimetry.

When using literature values of ΔH_s , be careful to distinguish between *enthalpy change* and *heat evolved*, which are opposite in sign. If the dissolution is exothermic, the amount of heat evolved is positive. However, the enthalpy change is negative (there is a net loss in system enthalpy). The table below lists values of ΔH_s for some common substances.

Molar Enthalpy of Solution for Selected Compounds
(in water at approx. 20°C, infinite dilution)

Compound	$-\Delta H_s$ (heat evolved) kcal/mole	Compound	$-\Delta H_s$ (heat evolved) kcal/mole	Compound	$-\Delta H_s$ (heat evolved) kcal/mole
Conc. HCl (11.7M)	2.9	RbOH	14.9	LiI	-14.9
HCl (gas)	17.9	CsOH	17.1	NaI	-1.6
Conc. HBr (8.9M)	1.8	NH ₃ (gas)	7.3	KI	-4.9
HBr (gas)	20.3	KHCO ₃	5.1	NaH ₂ PO ₄	-5.2
Conc. HNO ₃ (15.9M)	1.8	K ₂ CO ₃	-6.6	Na ₃ PO ₄	-13.0
Conc. HI (7.6M)	1.3	NaHCO ₃	4.1	Na ₂ S ₂ O ₆	5.8
Conc. HClO ₄ (11.7M)	1.0	Na ₂ CO ₃	-5.6	AgNO ₃	-5.4
Conc. C ₂ H ₄ O ₂ (17.4M)	0.5	AlCl ₃	-77.9	KMnO ₄	-10.4
HF (gas)	14.7	NH ₄ Cl	-3.5	CaCl ₂	19.3
H ₃ PO ₄	-2.8	NH ₄ NO ₃	-6.1	CaCl ₂ ·6H ₂ O	-3.9
LiOH	5.6	NaCl	-0.9	CuSO ₄	16.4
LiOH·H ₂ O	1.6	KCl	-4.1	CuSO ₄ ·5H ₂ O	-2.4
NaOH	10.6	KBr	-4.8	NaSO ₄	0.36
KOH	13.8	MgCl ₂	-36.3	NaSO ₄ ·10H ₂ O	-19.0

Sources: [106, 114, 153, 259]

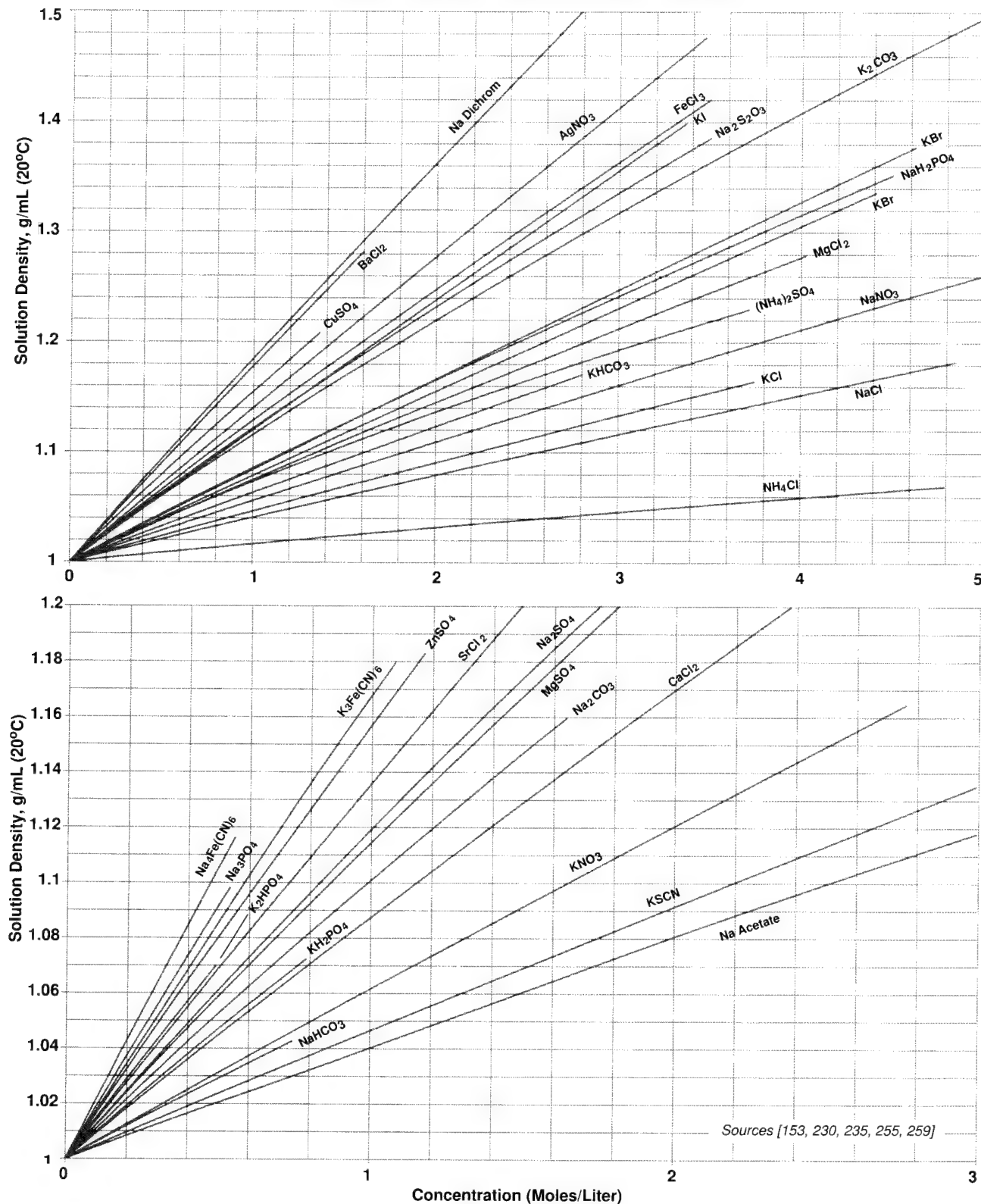


The chart above shows the densities of aqueous solutions of some common acids and bases as a function of molar concentration at room temperature (~20°C). Sources [153, 230, 235, 255, 259].

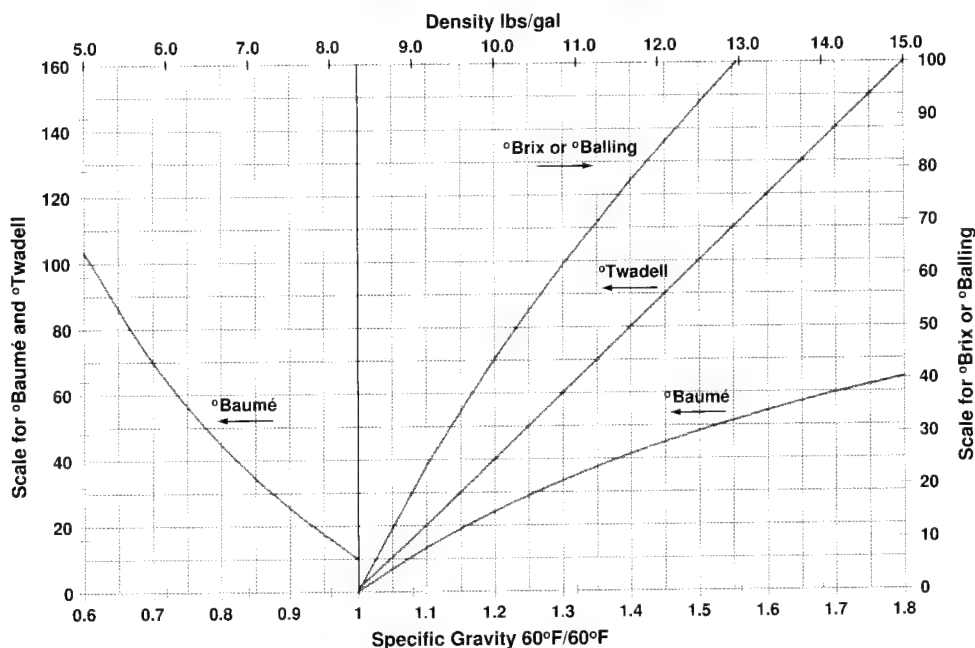
To make an approximate conversion between molar and weight percent solutions, use the densities read from the graph above and the following equation, where **M** = molarity and **mw** = the compound molecular weight:

$$\text{Wt \%} = \frac{M \times \text{mw} \times 0.1}{\text{density}}$$

Density of Selected Aqueous Inorganic Solutions



Comparison of Density Scales



Relationship of Various Density Scales to Specific Gravity

$^{\circ}\text{Baumé} = 145 - \frac{145}{\text{sg}}$ (Heavier than H ₂ O)	$^{\circ}\text{Baumé} = \frac{140}{\text{sg}} - 130$ (Lighter than H ₂ O)	$^{\circ}\text{Twadell} = \frac{\text{sg} - 1}{0.005}$	$^{\circ}\text{API} = \frac{141.5}{\text{sg}} - 131.5$
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The chart and table above compare several scales of density and specific gravity used industrially, most of which were originally developed for use with simple floating hydrometers. **Specific gravity**, often mistakenly called density, is the weight of a volume of liquid at a given temperature with respect to the weight of the same volume of a reference liquid at the same temperature. For the scales here, the reference liquid is water at 60°F, or 15.6°C (density of water at 60°F = 0.9990 g/cm³). Note that separate **°Baumé** scales exist for liquids heavier than water and for liquids lighter than water. **°Twadell** is a linear numbered scale related to specific gravity. **°API** scale was developed by the American Petroleum Institute for use with petroleum products. **°Brix** or **°Balling** are identical scales used in the food and brewing industries for expressing sugar concentration. Values are equal to the actual grams of cane sugar in 100 ml of solution. Sources [195, 200, 253, 267].

Comparison of Various Concentration Scales

$\text{Molar (M)} = \frac{\text{gmole of solute}}{\text{liter of solution}}$	1 ppb = 0.001 ppm
$\text{Normal (N)} = \frac{\text{M} \times \text{No. of ionizable groups}}{\text{liter of solution}}$	10 ppb = 0.01 ppm
$\text{Weight \%} = \frac{\text{g solute} \times 100}{\text{g solute} + \text{g solvent}}$	100 ppb = 0.1 ppm
$\text{Volume \%} = \frac{\text{liters solute} \times 100}{\text{liters of solution}}$	1000 ppb = 1 ppm
$\text{ppm} = \frac{\text{mg solute}}{\text{kg solution}} = \frac{\text{mg solute}}{\text{L water}}$	1 ppm = 0.0001 %
	10 ppm = 0.001 %
	100 ppm = 0.01 %
	1000 ppm = 0.1 %
	10000 ppm = 1 %
	100000 ppm = 10 %
	1000000 ppm = 100 %

Source: [176]

Grades of Chemical Purity

Technical Grade	Used industrially, but may be unsuitable for laboratory purposes because of high impurity levels. However, if this grade will be used at the manufacturing scale, it is wise to begin using it as early as practical in process development and scale-up.
Practical Grade	Lower levels of impurities, suitable for most laboratory purposes.
USP	Pure enough to pass certain tests prescribed by the US Pharmacopoeia. However, not all impurities or characteristics may be covered by the prescribed tests. Suitable for most laboratory purposes.
CP	"Chemically Pure". May be as pure as Reagent Grade, but the term is ambiguous and use may depend on the specific purpose and the effect of certain individual impurities.
Spectroscopic Grade	Meets the special requirements for solvent purity for spectrophotometry in the UV, IR and near-IR regions as well as for NMR and fluorometry. Very high order specifications for water content, evaporation residue and absorbance characteristics.
Chromatographic Grade	Minimum purity level of >99% (mole %) as determined by gas chromatography. Typically, no individual impurity exceeds 0.2%. Usually the chromatogram and specific test conditions accompany the chemical.
Reagent (or Analyzed Reagent) Grade	Certified to contain impurities below the specifications set by the ACS committee on Analytical Reagents. Each bottle is identified by batch or lot number. Useful for analytical work. Again, not all impurities or characteristics may be covered by the tests.
Primary Standard	Sufficiently pure that the substance may serve as a reference standard in analytical procedures. Trace impurities may vary depending on the manufacturer.

Source: [70]

Drying Agents for Solvents and Solutions

Where azeotropic drying is not a suitable alternative (see page 6-25), solvents and organic solutions are often dried by direct addition of a drying agent, usually an insoluble solid with a high capacity for absorbing water, which is then filtered off, or by passing the liquid through a bed of the agent. Drying agents should be selected carefully based on their capacity, reactivity and suitability for the specific task. Some dehydrating agents are powerful and can react violently if the water content is high. For example, CaCl_2 should not be used to dry alcohols, phenols, amines, amides, ketones or certain aldehydes and esters. Generally speaking, the agent should be added slowly, good agitation should be provided and a suitable time allowed for the agent to act. In some cases, more than one treatment may be necessary. Some common drying agents are listed below. Sources [70, 110, 232].


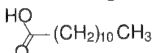






















Agent	gms water removed / gram agent	Relative Speed	Application / notes
Alumina (Al_2O_3)	0.20	v. fast	Hydrocarbons (HCs)
BaO	0.12	-	HCs, aldehydes, alcohols
CaCl_2	0.15	v. fast	Inert organics (HCs and Alkyl halides - may react with N- or O-containing compounds)
CaH_2	0.85	v. fast	Potent drying agent for hydrocarbons, ethers, esters, alcohols. Evolves H_2
CaO	0.31	-	Ethers, esters, alcohols, amines
CaSO_4 (drierite)	0.07	v. fast	Most organics
H_2SO_4	v. high	v. fast	Alkanes, aromatics, and halides
K_2CO_3	0.26	fast	Alcohols, esters, ketones and nitriles. Avoid acidic compounds
KOH	v. high	fast	Amines and inert solvents in which KOH is insoluble
MgSO_4	1	fast	Excellent for most organics. Inert to slightly acidic, may dissolve in some solvents
Molecular Sieves 3A	0.18	fast	Very efficient. Pore size 3\AA , also absorbs NH_3 .
Molecular Sieves 4A	0.18	fast	Very efficient. Pore size 4\AA , also absorbs CO_2 , SO_2 , HS, ethane, ethylene, propylene, and ethanol
Na_2SO_4	1.25	slow	Very mild, slow acting but very high capacity
NaOH	v. high	fast	Amines and inert solvents in which NaOH is insoluble
Silica Gel	0.2	v. fast	Hydrocarbons

Chemical Nomenclature
















Names of Common Ions

Acetate	$\text{C}_2\text{H}_3\text{O}_2^-$	Chlorite	ClO_2^-	Ferrous	Fe^{+2}	Perchlorate	ClO_4^-
Ammonium	NH_4^+	Chromate	CrO_4^{2-}	Fluoride	F^-	Periodate	IO_4^-
Bicarbonate	HCO_3^-	Chromic	Cr^{+3}	Hydroxide	OH^-	Permanganate	MnO_4^-
Bisulfate	HSO_4^-	Chromous	Cr^{+2}	Hypochlorite	ClO^-	Phosphate	PO_4^{3-}
Bisulfide	HS^-	Cupric	Cu^{+2}	Iodide	I^-	Phosphite	PO_3^{3-}
Bisulfite	HSO_3^-	Cuprous	Cu^+	Mercuric	Hg^{+2}	Stannic	Sn^{+4}
Bromate	BrO_3^-	Cyanate	OCN^-	Mercurous	Hg^+	Stannous	Sn^{+2}
Bromide	Br^-	Cyanide	CN^-	Neckelic	Ni^{+3}	Sulfate	SO_4^{2-}
Bromite	BrO_2^-	Dichromate	$\text{Cr}_2\text{O}_7^{2-}$	Nickelous	Ni^{+2}	Sulfide	S^{2-}
Carbonate	CO_3^{2-}	Ferric	Fe^{+3}	Nitrate	NO_3^-	Sulfite	SO_3^{2-}
Chlorate	ClO_3^-	Ferricyanide	$\text{Fe}(\text{CN})_6^{3-}$	Nitrite	NO_2^-	Thiocyanate	SCN^-
Chloride	Cl^-	Ferrocyanide	$\text{Fe}(\text{CN})_6^{4-}$	Perbromate	BrO_4^-	Thiosulfate	$\text{S}_2\text{O}_3^{2-}$

Principle Functional Groups in Organic Chemistry

Group Name	Example		Group Name	Example	
Alkanes (Paraffins)	Ethane	$\text{H}_3\text{C}-\text{CH}_3$	Carboxylic Acids	Acetic Acid	
Alkenes (Olefins)	Ethylene	$\text{H}_2\text{C}=\text{CH}_2$	Fatty Acids	Lauric Acid	
Alkynes	Acetylene	$\text{HC}\equiv\text{CH}$	Acyl Halides	Acetyl Chloride	
Dienes	1,3-Butadiene		Acid Anhydrides	Acetic Anhydride	
Arenes (Aromatics)	Benzene		Esters	Ethyl Acetate	
Alkyl Halides	Chloroethane		Amides	N, N-Dimethylacetamide	
Alkenyl Halides	Vinyl chloride		Amines, primary	Ethylamine	
Aryl Halides	Chlorobenzene		Amines, secondary	Diethylamine	
Alcohols	Ethanol		Amines, tertiary	Triethylamine	
Phenols	Phenol		Nitriles	Acetonitrile	
Ethers	Diethyl ether		Nitro Compounds	Nitrobenzene	
Epoxides	Ethylene oxide		Thiols	Ethanethiol	
Aldehydes	Acetaldehyde		Sulfides	Diethyl Sulfide	
Ketones	Acetone				

Structures of Other Common Organic Substituents

Acetyl		Butyl or <i>n</i> -Butyl		Neopentyl	
Allyl		Carbonyl		Phenyl	
Amino	$\text{H}_2\text{N}-\text{R}$	Hydroxyl	$\text{R}-\text{OH}$	Propyl or <i>n</i> -Propyl	
Benzoyl		Isobutyl		<i>sec</i> -Butyl	
Benzyl (Phenylmethyl)		Isopropenyl		<i>tert</i> -Butyl	
		Isopropyl		Vinyl (Ethenyl)	

Adapted from [46, 261]

Some Common Types of Organic Reactions

Reaction	Example	Notes
Alkylation	$R-NH_2 + 2 CH_3OH \longrightarrow R-N \begin{matrix} CH_3 \\ \\ CH_3 \end{matrix} + 2 H_2O$	Addition of an alkyl group, in this case, to a secondary amine by reaction with an alcohol.
Amination	$R-Cl + 2NH_3 \longrightarrow R-NH_2 + NH_4^+ Cl^-$	Addition of an amine group. Shown here, the amination of an alkyl halide with ammonia.
Condensation	$2 RCH_2OH \longrightarrow RCH_2OCH_2R + H_2O$	Two molecules join to form a large molecule and liberate a small one. Here two alcohols condense to form an ether and eliminate water.
Dehydration	$CH_3-CH_2OH \longrightarrow CH_2=CH_2 + H_2O$	Characterized by the elimination of water, for example when an alcohol converts to an alkene.
Dehydrogenation	$CH_3-CH_3 \longrightarrow CH_2=CH_2 + H_2$	Characterized by the elimination of H_2 . The conversion of ethane to ethylene shown here requires extremely high temperature.
Esterification	$R-OH + R'-\overset{\overset{O}{\parallel}}{C}OH \longrightarrow R'-\overset{\overset{O}{\parallel}}{C}OR + H_2O$ $R-OH + R-\overset{\overset{O}{\parallel}}{C}Cl \longrightarrow R-\overset{\overset{O}{\parallel}}{C}OR + HCl$	Formation of an ester from an alcohol and a carboxylic acid (Fischer esterification, often driven forward by azeotropic removal of water) or from alcohol and an acyl halide. Usually acid catalyzed.
Formylation	$CH_2=CH_2 + CO \xrightarrow{H_2} CH_3-CH_2-\overset{\overset{O}{\parallel}}{C}H$	Shown here is the hydroformylation of an alkene to an aldehyde. Usually catalyzed by a noble metal.
Friedel-Crafts	$\text{C}_6\text{H}_6 + R-Cl \xrightarrow{AlCl_3} \text{C}_6\text{H}_5-R + HCl$ $\text{C}_6\text{H}_6 + R-\overset{\overset{O}{\parallel}}{C}-Cl \xrightarrow{AlCl_3} \text{C}_6\text{H}_5-\overset{\overset{O}{\parallel}}{C}-R + HCl$	Shown here are Friedel-Crafts alkylation and acylation of benzene, using an alkyl halide and acyl halide, respectively. Friedel-Crafts reactions are usually catalyzed by aluminum chloride.
Halogenation	$\text{C}_6\text{H}_6 + Br_2 \longrightarrow \text{C}_6\text{H}_5-Br + HBr$	Addition of a halogen, for example, the bromination of benzene. Usually requires a catalyst, in this case Fe.
Hydration	$CH_2=CH_2 + H_2O \longrightarrow CH_3-CH_2-OH$	The addition of water. In this example water is added to an alkene to form an alcohol.
Hydrolysis	$R-Br + H_2O \longrightarrow R-OH + HBr$	Water is consumed, in this case, to form an alcohol from an alkyl halide.
Hydrogenation	$CH_2=CH_2 + H_2 \longrightarrow CH_3-CH_3$	Hydrogenation is often carried out at high temperature and pressure in the presence of a noble metal catalyst.
Nitration	$\text{C}_6\text{H}_6 + HNO_3 \longrightarrow \text{C}_6\text{H}_5-NO_2 + H_2O$	Addition of a nitrate group, for example by treatment of benzene with HNO_3 in the presence of H_2SO_4 .
Oxidation	$R-OH \longrightarrow R-\overset{\overset{O}{\parallel}}{C}H \longrightarrow R-\overset{\overset{O}{\parallel}}{C}-OH$	Oxidation of an alcohol to an aldehyde or of an aldehyde to a carboxylic acid. Often carried out using an oxidizing agent such as ammonium dichromate (Na_2CrO_2) and appropriate catalysts.
Sulfonation	$\text{C}_6\text{H}_6 + H_2SO_4 \longrightarrow \text{C}_6\text{H}_5-SO_3H + H_2O$	Addition of a sulfonate group, for example by treatment of benzene with H_2SO_4 .

Sources; [46, 261]

The table above lists some fundamental classes of reactions encountered in organic chemistry. Some of the examples given are not practical laboratory synthetic pathways because they may require extreme temperatures, pressures, or special catalysts. Also, many of the reactions are not limited to the functional groups shown in the examples, but can act with numerous chemical groups. For example, hydrogenation is commonly carried out on aldehydes, ketones, esters, alkynes and many other species, yielding products specific to the substrates used. Many may also be equilibrium reactions. The examples are simply included here to illustrate the principle and typical end-products of the reaction type.

9 Chemical Hygiene and Safety

Contents

CHEMICAL SAFETY

General Notes on the Safe Handling of Chemicals	9-2
The Chemical Hygiene Plan	9-3
Material Safety Data Sheets	9-4
Classification of Hazardous Substances	9-5
Toxicity and Health	9-6
Fire Safety	9-6
Fire Extinguishers	9-7
Chemical Labeling	9-8
List of Incompatible Chemicals	9-9
Waste Effluent Disposal	9-10

PERSONAL PROTECTIVE EQUIPMENT

Eye and Face Protection	9-12
Hearing Protection	9-12
Hand Protection	9-13
Glove Selection Guide	9-13
Respiratory Protection	9-16

ELECTRICAL SAFETY – See Chapter 5.

SAFE HANDLING OF SOLVENTS – See Chapter 6.

SAFE HANDLING OF COMPRESSED GASES – See Chapter 8.

General Notes on the Safe Handling of Chemicals

Toxic, corrosive, reactive, flammable or otherwise dangerous chemicals are everywhere in the chemical facility. Hundreds of thousands of compounds exist, and it is impossible to know the properties of all of them. However, it is the worker's responsibility to know as much as possible and to follow practices that help protect his or her own safety as well as the safety of others. All companies should have their own guidelines for handling chemicals, but here are just a few common sense principles that always apply. More information on specific aspects of chemical safety are found throughout the chapter, including types of hazards, exposure limits, and selection of protective equipment.

Know the Hazards – Never attempt to run a chemical process without knowing the chemical and toxic properties of all materials involved. Read the MSDS for compounds with which you are not familiar and be prepared to handle contingencies. Remember that while intermediates may not be listed, they may still be pharmacologically active or toxic.

Use Protective Equipment – Always use proper personal protective equipment such as Z87 eye protection, respirators, gloves, face shields, ear protection, hard hats, etc. Know the safety rules applied to their use. Know the location of emergency equipment such as fire extinguishers, safety showers, and eyewash fountains.

Wear Proper Attire – Do not wear baggy or loose fitting clothing. Keep long hair tied back. Wear appropriate footwear, including steel-toe safety shoes as required.

Practice Good Chemical Hygiene – Never bring food or drink into or smoke in a laboratory or plant environment. Never touch chemicals with bare hands. Decontaminate and change or remove lab attire after working with chemicals. Dispose of gloves immediately after exposure.

Avoid Exposure – Chemical must be handled in such a way that they do not escape into the environment. Never work with chemicals outside of a fume hood when the possibility of releasing toxic materials or gases exists. Ensure that there is adequate ventilation. Use secondary containment as required.

Transport Carefully – Always transport chemicals with a secondary container. Avoid moving chemicals in an elevator. Move 55-gallon drums only with the proper equipment – do not roll! When transferring liquid chemicals, ground and bond (wire together) drums and containers. Check that containers and hoses are in good condition.

Store Chemicals Properly – Never store chemicals in unlabeled containers. Never use chemicals from unlabeled containers. Never return excess chemicals to their original containers. Don't leave containers open when not in use. Do not use fume hoods to store chemicals, as it may disrupt the air flow pattern. Store acids and bases separately as well as oxidizers and flammables. Do not store chemicals next to each other without knowing it is safe to do so (read the MSDS or see the list of incompatible chemicals on page 9-9). Keep the minimum amount necessary in the plant or lab. Adhere to the appropriate Building Code guidelines for storage limitations of hazardous and flammable substances. Maintain a current chemical inventory of the lab. Secure all gas cylinders.

Develop Good Work Habits – Never work alone. Never leave chemical reactions unattended without notifying someone. Clean up or put away equipment after use. Follow company policies with regard to using hazardous reagents. Clean up spills immediately.

Know How to Respond – Report all spills and accidents immediately. Do not hose down spills with water unless you know it is safe to do so. Know what kinds of fire extinguishers can be used for each situation. Follow company policy in regard to fire fighting and emergency response.

Practice Safe Disposal – Dispose of wastes promptly. Comply with local regulations concerning storage limits and segregation of wastes (see page 9-10). Never flush waste streams or chemicals down the drain.

Be Observant – Be aware of your surroundings and periodically check around for leaks, puddles, bulging containers or hissing sounds, which could indicate a dangerous situation. Pay attention to unusual odors that might indicate the release of a toxic substance, although many substances have no strong odor, and olfactory fatigue can limit your ability to smell it if you are exposed to it for a time. Pay attention to any unusual symptoms, which could indicate exposure.

The Chemical Hygiene Plan

Many small companies may find that it takes a great deal of time to get all of their “ducks in line” to meet the requirements of the various regulatory agencies that oversee the CPI. But safety is not the area to cut corners. Developing and *adhering to* a comprehensive, workable Chemical Hygiene Plan (CHP) is one of the most important steps a company can take. This should be a written document, outlining in detail the principles and practices that the company has adopted for ensuring the safety of their operations, with the full buy-in of company management all the way up to the CEO. A Chemical Hygiene Manual should be made available to all employees so that they are aware of company policies. The plan should also be reviewed and updated as necessary at regular intervals.

Personnel safety must always be the number one priority in any chemical operation, followed by preservation of capital equipment and compliance with local, state and federal environmental laws. But a good CHP can do much more than that. Through regular training and by raising worker awareness, the plan will inevitably result in safer work habits, better communication, more streamlined operations, decreased downtime, fewer mistakes and an improved bottom line.

Here are some of the elements that a comprehensive CHP should address:

Employee Training Program – requiring regularly scheduled (annual) safety training sessions for all employees involved in handling chemicals. This should include use of personal protective equipment, rules and operating procedures. A system should be in place for maintaining training records. New employees should receive a safety orientation and be made aware that working safely is a condition of employment.

Emergency Response Plan – procedures for dealing with accidents, spills, fires. The plan should include a list of emergency coordinator contact information, periodic evacuation drills, fire extinguisher training, policies about reporting accidents, HAZWOPER training and site maps including information about evacuation routes in case of emergency.

Waste Disposal Plan – this plan should be in accordance with all RCRA and local regulations and include personnel training, record keeping and document retention.

Facility Inspections and Environmental Monitoring – periodic testing of fume hoods, roof fans, and room ventilation systems for proper operation, and testing for possible contamination.

Lab or Plant Inspection Program – including periodic inspections to help ensure that chemicals are stored and labeled properly, safety equipment is available and in working order, that access ways are clear and equipment is not blocked or crowded, appropriate personal protective equipment is in use, and other safety policies are observed.

First Aid Training Program – including CPR and emergency medical treatment for chemical exposure.

Safety Committee – establish a safety committee representing various departments with regularly scheduled meetings to address safety concerns and improve safety programs. A rotating schedule should involve all employees.

Lockout-Tagout – a lockout/tagout and training program to ensure that workers follow the proper procedures for disconnecting energy sources from equipment before working on it, and that covers contractor safety, shift changes, etc.

Process Safety Review – should include a policy for obtaining safety information (calorimetry studies, etc.) and a system for conducting Haz-Op (Hazards and Operability) reviews for new processes and criteria for proceeding with scale-up operations.

Chemical Inventory – electronic inventory of all chemicals in the facility with expiry information, and including policies for procurement, receiving and storage of chemicals.

Contractor and Visitor Safety – including policies to ensure that outside contractors working in your facility are aware of the hazards, procedures, lockout/tagout policies and required permits for performing hotwork and other tasks for their own safety and that of everyone else working at the site.

Safety Library – establishment of a central location for books on safety, MSDSs, lab safety handbooks, safety equipment catalogs, etc. All should be encouraged to be familiar with the resources kept there.

Material Safety Data Sheets

The Material Safety Data Sheet (MSDS) is the industry standard for compiling the safety information for chemicals and other hazardous substances. An MSDS must be available for every chemical used or produced in your facility. While sometimes difficult to decipher at first, it should be a primary source of information and the basis for discussions of required handling techniques and potential hazards. Data on compatibility with other chemicals can be valuable in the design of safe experimental procedures, and first aid and response measures can prepare users for most contingencies.

Suppliers of chemicals are required to send a copy of the MSDS along with every shipment of a compound to a facility, unless it is a substance ordered on a regular basis. Copies of the MSDSs for all compounds being used in the lab or pilot plant should be kept on hand in a readily accessible location. MSDSs should be prepared for proprietary intermediates and other unlisted compounds, particularly if being shipped off-site.

While there is no strictly set format, MSDSs are required by law to include certain key information, and other information is also often included. A well-written MSDS will usually consist of the following sections:

Chemical Name – IUPAC names or common names.

Synonyms – abbreviations, acronyms and industry or trade names, if applicable.

CAS Registry Number – a unique identifying number registered in Chemical Abstract Services, Inc. database.

Manufacturer Identification – name and locations of manufacturer, including emergency contact information.

Preparation or Revision Date – date on which MSDS was issued or revised.

Composition – lists all ingredients present in an amount over 1% (carcinogens over 0.1%), identified by chemical name.

Physical Property Data – includes information such as appearance, odor, boiling point, melting point, vapor pressure, density, evaporation rate (usually relative to n-butyl acetate), etc.

Fire and Explosion Hazard Data – includes information such as flashpoint, autoignition temperature, explosive limits in air, recommended fire extinguishing media and fire fighting measures.

Reactivity Hazard Data – stability, reactivity with water, and a list of other incompatible chemicals.

Health Hazard Data – toxicological information, LD50 or LC50, carcinogenic or mutagenic potential, likely methods of exposure (inhalation, ingestion, skin absorption). Should also include signs and symptoms of exposure.

Exposure Limits – these may include OSHA permissible exposure limit (PEL) and/or ACGIH threshold limit value (TLV) usually in ppm or mg/m³. See page 9-6 for more on the definition and application of these terms.

Handling and Storage Precautions – special storage requirements, list of compounds from which to be separated.

Recommended Personal Protective Equipment – respirator and glove selection, eye protection and protective clothing, and other exposure controls such as ventilation requirements.

Accidental Release Response Measures – appropriate equipment and procedures for cleaning up a spill or other release

First Aid Measures – actions to take upon exposure to minimize possible injury (induce vomiting, flush with cold water, remove to fresh air, etc.).

Ecological Information and Disposal Considerations – recommended means of disposal, precautions for disposal.

Transport Information – any limitations or precautions for packaging and shipping safely, DOT shipping information, UN hazard class, packaging group, etc.

Regulatory Information – any special considerations based on classification under TSCA, OSHA, Clean Water Act, etc.

Other Information – any other information applicable to safe handling and use.

When preparing MSDSs for new compounds, be sure to use clear terminology and identify the hazards properly. Model yours after the example of a professionally prepared MSDS. There are a number of excellent sources for MSDSs, including the Aldrich Chemical Company MSDSs CD-Rom, which lists nearly 500,000 compounds and the free resource maintained by the U.S. Government at the SIRI Safety Information website <http://hazard.com/msds/>.

Classification of Hazardous Substances

The following terms and classifications are based on applicable NFPA, CGA, NESC, and OSHA codes.

<p>Flammable Liquids (also called Class I Liquids)</p> <p>Liquids having a flash point below 100°F (37.8°C) and a vapor pressure less than 40 psia at 100°F (37.8°C)</p> <p>Class IA: Flashpoint below 73°F (22.8°C) and boiling point below 100°F (37.8°C) - examples: ethyl ether, pentane, ethylamine</p> <p>Class IB: Flashpoint below 73°F (22.8°C) and boiling point at or above 100°F (37.8°C) - examples: acetone, acetonitrile, toluene</p> <p>Class IC: Flashpoint between 73°F (22.8°C) and 100°F (37.8°C) - examples: butanol, DMSO, nitrobenzene</p>
<p>Combustible Liquids</p> <p>Liquids having a flash point at or above 100°F (37.8°C)</p> <p>Class II: Flashpoint between 100°F (37.8°C) and 140°F (60°C) - examples: anisole, cyclohexanone, DIBK</p> <p>Class IIIA: Flashpoint between 140°F (60°C) and 200°F (93°C) - examples: benzaldehyde, cyclohexanone, methylcarbitol</p> <p>Class IIIB: Flashpoint at or above 200°F (93°C) - examples: mineral oil, propylene glycol, triethanolamine</p>
<p>Flammable Gases</p> <p>Gases which are flammable in a mixture of 13% or less (by volume) with air, or with a flammability range of more than 12%</p>
<p>Non-Flammable Gases</p> <p>Compressed gases which are not flammable. Examples include ammonia, argon, helium, nitrogen, etc.</p>
<p>Flammable Solids</p> <p>Solids liable to cause a fire due to friction, absorption of moisture, spontaneous chemical change, retained heat, or that can be readily ignited and burn vigorously enough to create a serious fire hazard</p>
<p>Corrosive Materials</p> <p>Materials that cause visible destruction or permanent alteration of living tissue. Subdivided as acids, bases and other corrosives</p>
<p>Organic Peroxides</p> <p>Organic compounds that contains a double oxygen (-O-O-) structure. These compounds are capable of causing a fire or detonating because of sensitivity to shock or upon decomposition to unstable compounds over time</p> <p>Class I: capable of deflagration (bursting into flames) but not detonation (concussive explosion)</p> <p>Class II: capable of burning rapidly and creating a severe reactivity hazard</p> <p>Class III: capable of burning rapidly and creating a moderate reactivity hazard</p> <p>Class IV: organic peroxides which burn in the same manner as ordinary combustibles and present minimal reactivity hazard</p> <p>Class V: not capable of sustaining combustion and present no reactivity hazard</p>
<p>Oxidizers</p> <p>Liquid or solid compounds that can initiate or promote combustion of other materials with which they come into contact, either in and of themselves, or by the release of oxygen or other oxidizing gases</p> <p>Class I: an oxidizer whose primary hazard is that it may increase the burning rate of other materials</p> <p>Class II: an oxidizer that may increase the burning rate of, or cause spontaneous ignition of, other materials</p> <p>Class III: an oxidizer that will severely increase the burning rate of other materials, or that may undergo vigorous decomposition on its own when catalyzed or exposed to heat</p> <p>Class IV: an oxidizer that can undergo an explosive reaction when catalyzed, shocked or exposed to heat</p>
<p>Poisons A (poisonous gases)</p> <p>Compressed gases so poisonous that a very small amount mixed with air is dangerous to life</p>
<p>Poisons B</p> <p>Liquids, solids or semisolids which are known to be toxic to humans, or that fall into one of the animal test categories below</p> <p>Oral Toxicity - LD50 in rats is 50 mg/kg body weight by a single oral dose</p> <p>Inhalation Toxicity - LD50 in rats occurs with one hour exposure time at 2mg/liter of air or less as vapor, dust or mist</p> <p>Skin Absorption - LD50 in rats occurs with continuous 24-hour exposure to bare skin of 200mg/kg body weight or less</p>
<p>Irritating Materials</p> <p>Liquids or solids which when exposed to air or fire give off dangerous or irritating fumes</p>
<p>Other Regulated Materials, Class A (ORM-A)</p> <p>Materials which have anesthetic, irritating, noxious or toxic properties not covered by other hazardous material categories</p>

Toxicity and Health

Toxic substances abound in the chemical industry. Understanding their effects is the first step towards their safe use. Chemicals can enter the body in four main ways: **inhalation**, **ingestion**, **absorption** through the skin (or eyes), or **injection** (accidental puncture, etc.). The effects of toxins can occur immediately or may not be noticed for a considerable length of time. **Acute poisoning** is typically due to exposure to a single large dose with immediate severe effects, whereas **chronic poisoning** is the result of prolonged or repeated exposure to cumulative poisons over days, weeks, months, or years. Often, the combined effects of two or more toxic substances can be much worse than predicted based on the properties of the individual substances.

Chemical exposure limits are guidelines established by OSHA, NIOSH and ACGIH to help employees and employers understand the hazards of the chemicals they handle. One such guideline is the amount of a given substance to which a worker may safely be exposed. A number of terms, commonly used for various situations, are explained below. Note that values vary from country to country.

The **threshold limit value (TLV)**, recommended by ACGIH, is the concentration to which workers may be continually exposed, day after day (based on a time-weighted average 8-hour workday or 40-hour workweek) without adverse effects. The **permissible exposure limit (PEL)** is a legal standard issued by OSHA essentially identical to the TLV. The **short term exposure limit (STEL)** is the highest allowable concentration to which workers may be exposed for a period of 15 minutes without experiencing irritation, tissue change, or enough impairment of function to compromise safety.

Ceiling – The concentration to which the worker cannot be exposed for even an instant. Above this concentration, corrective action must be taken or protective equipment must be worn.

Immediately Dangerous to Life or Health (IDLH) – an atmosphere that poses an immediate hazard to life or causes an immediate irreversible debilitating effect on the worker's health.

The **toxic concentration (TxC)** is the concentration of a substance in air or solution known to produce harmful effects in humans. The **toxic dose (TxD)** is the dose of a chemical that has been reported to produce harmful, but not fatal, effects in humans. The **lethal dose 50 (LD50)** refers to the dose of a toxic substance that is expected to kill half of a population of test animals by exposure means other than inhalation. For inhalation hazards, the **lethal concentration 50 (LC50)** is used to express the concentration expected to kill half of a population of test animals after a 4-hour exposure.

The terms **approximate lethal dose (LDca)**, **lethal dose (LD)** and **minimum lethal dose (MLD)** are all used at various times to express the dose of a toxic substance that has been reported to (or is expected to) cause death in humans. The **maximum tolerated dose (MTD)** is the maximum amount of a substance that can be tolerated without death. The **approximate lethal concentration (LCca)** refers to the concentration of a substance that can be expected to cause death by inhalation during an exposure period of one day.

Some other important terms include: **carcinogen** (suspected or known to cause cancer), **mutagen** (can cause genetic damage), **teratogen** (can cause physical defects in developing fetus), and **lachrymator** (has an irritating or burning effect on eyes or respiratory tract).

Fire Safety

The fire hazards associated with flammable liquids, gases and other substances are generally well characterized. As a flammable liquid is heated, the vapor pressure increases, raising the concentration of vapors in the air above the liquid surface to a point where they can be ignited by a spark or flame. The vapor concentration may not be high enough to sustain combustion, so ignition takes the form of only a momentary flash. The liquid temperature at which the vapor mixture just becomes ignitable is called the **flash point**. If no ignition source is present, the liquid can be heated further until the vapor concentration will render a self-sustaining flame if ignited. If the liquid is heated to a high enough temperature, it will spontaneously ignite. This is called the **auto-ignition temperature**.

In the air/vapor mixture, if the vapor concentration is too low, it cannot be ignited. If it is too high, it likewise cannot be ignited because there is insufficient oxygen present to allow combustion. The minimum and maximum possible vapor concentrations that are ignitable are called the **explosive limits in air**, usually expressed as volume %. The vapor pressure

and equilibrium concentration in air are related as follows:

$$\text{volume \%} = \frac{\text{vapor pressure}}{\text{atmospheric pressure}}$$

Thus, if a liquid has a vapor pressure of 76 mm Hg, its concentration is $76/760 = 10 \text{ vol\%}$. The vapor pressure of flammable gases is usually very high, and most form explosive mixtures in air at room temperature.

Some substances can self-ignite even in the absence of an ignition source in a process known as **spontaneous combustion**. This is caused by heat generated in the substance due to chemical degradation or reaction with air during storage. Many non-petroleum based oils, charcoal dust and grain dusts are known to generate heat on storage and are therefore susceptible to spontaneous combustion.

Flashpoint, auto-ignition temperature, explosive limits and other fire hazard information are listed in product MSDSs. Information on the safe use of flammable solvents can be found in Chapter 6. NFPA flammability codes and the use of flammables in hazardous locations are discussed in Chapter 5.

Fire Extinguishers

Fire extinguishers should be selected based on the types of fires they may be used to fight (see below). They must be located properly (in plain sight near an exit, and away from potential fire hazards). They must be kept in good working order, inspected periodically by a certified dealer, and recharged after *any* use. Local fire departments usually provide employee training sessions on the proper care and use of extinguishers, and these are highly recommended.

Always adhere to your company's policy concerning fire-fighting, which usually reads something like this: *Immediately sound the alarm to evacuate the building and notify authorities. Then, if the fire is small and you feel that you can extinguish it quickly and safely, you may attempt to do so if you wish. If your initial attempt is not immediately successful, stop, leave the area and close the door behind you.* It is foolish to attempt extinguishing fires without the proper respiratory and other protective equipment, since fire and heat can spread so rapidly and noxious fumes can overcome the unwary in seconds. All of the above assumes that you are familiar with the various extinguisher types and know exactly where they are and how to use them. If there is any doubt in your mind, do not make the attempt.

For purposes of fire-fighting, the NFPA has categorized fires into four classes as shown in the table below along with the extinguishers recommended for each type of fire. All approved fire extinguishers will be clearly labeled with the type or types of fires (by code letter) for which they are suitable. It is often necessary to have more than one type of extinguisher on hand to face all likely contingencies.

Water extinguishers usually contain sodium bicarbonate and sulfuric acid to propel the water towards the fire. These extinguisher have very limited utility. **Carbon dioxide (CO₂)** extinguishers are effective against types B and C fires, but be careful of the high pressure and the possibility of asphyxiation in confined spaces. **Dry chemical** (ammonium phosphate or sodium bicarbonate) are very effective at smothering types A, B and C fires but leave a residue that may be difficult to clean from delicate equipment. **Met-L-X** extinguishers are specifically designed for burning metals and are not particularly effective against other types of fires. **Halon** (halogenated hydrocarbon) extinguishers are quite effective against A, B and C type fires, but are ozone-depleting and the danger of asphyxiation in confined areas also exists.

Classes of Fires and Recommended Extinguishers

Fire Class	Description	Recommended Extinguisher
A	Common combustibles - wood, cloth, paper, rubber, plastics and the like	Water, Dry Chemical, Halon
B	All flammable liquids - NFPA Class I, II & III liquids, gasoline, oils, greases, paints, etc. For flammable liquid classifications, see Chapter 6.	CO ₂ , Dry Chemical, Halon
C	All electrical equipment fires, or fires involving live electrical systems including wiring, circuit breakers, etc.	CO ₂ , Dry Chemical, Halon
D	All fires involving combustible metals or metal dusts - Na, Mg, K, Pd, lithium aluminum hydride, etc.	Met-L-X or dry sand

Sources: [25, 175]

Chemical Labeling

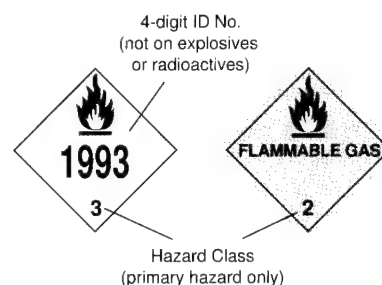
Chemical container labels are intended to display information on the identity of the substance and its potential hazards, manufacturer contact information, recommended personal protective equipment and other information. Labeling standards and requirements are established by several agencies, primarily the Department of Transportation (DOT) and the NFPA, but also OSHA, ANSI, the HMIS and the ISO. Sources [25, 146, 175].

DOT – DOT diamond-shaped labels are required on all shipped packages containing hazardous materials and wastes. Larger DOT placards must be visible on all bulk packages, freight containers, rail cars, etc. Strict guidelines specify the size, color, appearance and content of DOT labels. Examples of some typical DOT labels are shown below along with a table indicating the 9 classes of hazardous compounds recognized by the DOT and some related labeling standards. These standards are documented in CFR49, Part 171-173 which includes a Hazardous Materials Table (HMT) listing all recognized hazardous materials, their required labels and four-digit hazardous materials identification numbers.

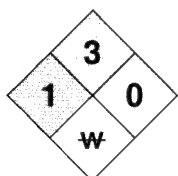
DOT Hazardous Materials Classification System

Class	Description	Label Colors
1	Explosives (subdivided as 1.1 – 1.6 based on severity)	Orange
2	Gases (Flammable, Non-Flammable, Poison and Toxic)	varies
3	Flammable Liquids	Red
4	Flammable Solids	varies
5	Oxidizers and Organic Peroxides	Yellow
6	Poisonous and Toxic	White
7	Radioactives	Yellow and White
8	Corrosives	Black and White
9	Miscellaneous Dangerous Goods	Black and White

Typical DOT Shipping Labels



NFPA – The National Fire Protection Association developed the familiar four-color diamond hazard identification system used for chemicals. Each colored section of the diamond represents a different hazard labeled with the numbers 0-4 to rate the severity of the hazard (4 being the most severe). The diamond, and detailed explanations of the hazard types and the meaning of the ratings are shown below.



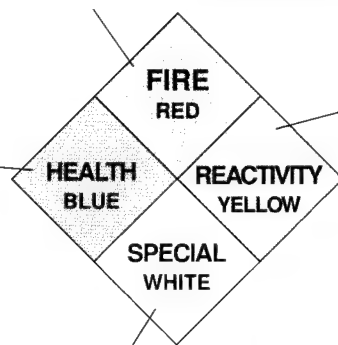
Example

HEALTH

- 0. No unusual hazard
- 1. CAUTION - May cause irritation
- 2. WARNING - May be harmful if inhaled or absorbed
- 3. HAZARDOUS - Corrosive or toxic if inhaled or absorbed, can cause serious injury
- 4. DEADLY - May be fatal upon even short-term exposure

FIRE

- 0. Will not burn
- 1. Combustible if heated (Flashpoint > 200°F)
- 2. CAUTION - Must be heated or exposed to high temperatures to ignite (Flashpoint < 200°F)
- 3. WARNING - Can be ignited under most ambient temperature conditions (Flashpoint < 100°F)
- 4. DANGER - Will rapidly vaporize or disperse in air at ambient conditions and burn readily (flammable gases and highly flammable liquids, Flashpoint < 73°F)



REACTIVITY

- 0. Normally stable, not water reactive
- 1. CAUTION - May react mildly if heated or mixed with water
- 2. WARNING - Unstable, may react violently with water
- 3. DANGER - May react explosively if shocked, heated under confinement, or contacted with water
- 4. DANGER - May detonate or explode at ambient conditions

SPECIAL



- Avoid Water

- Radioactive

ACID - Acid

ALK - Alkali

COR - Corrosive

OXY - Oxidizing Chemical

HMIS – Also widely used are Hazardous Materials Identification System labels, which use the same color codes as the NFPA system, but in rectangular form, and with severity ratings with nearly identical meaning.

Incompatible Chemicals

Substance	Avoid Contact With or Storage Near:
Acetic acid	Chromic acid, nitric acid, NaOH, KOH, ethylene glycol, perchloric acid, peroxides, permanganates
Acetone	Concentrated nitric and concentrated sulfuric acid
Acetylene	Chlorine, bromine, copper, silver, fluorine, mercury
Ammonia (anhydrous)	Mercury, chlorine, calcium, hypochlorite, iodine, bromine, HF, mineral acids
Ammonium nitrate	Acids, metal powders, chlorates, sulfur, flammable liquids
Aniline	Nitric acid, hydrogen peroxide
Azides	Acids (generate hydrogen azide)
Bromine	Anhydrous ammonia, acetylene, hydrogen, petroleum gases, turpentine, benzene, sodium carbide
Calcium hypochlorite	Acids, activated carbon, water
Calcium metal	Water, carbon dioxide, chlorinated hydrocarbons, halogens
Carbon (activated)	Calcium hypochlorite, oxidizing agents, unsaturated oils
Cesium metal	Water, carbon dioxide, chlorinated hydrocarbons, halogens
Chlorine	Ammonia, acetylene, butadiene, petroleum gases, hydrogen, hydrocarbons, sodium carbide, turpentine, benzene, metal powders
Chlorine dioxide	Carbon monoxide, mercury, ammonia, methane, phosphine, hydrogen sulfide
Chlorosulfonic acid	Water, metal powders, alcohols, ammonia, esters, hydrogen peroxide
Chromic acid	Acetic acid, acetic anhydride, camphor, glycerine, turpentine, alcohol
Copper	Acetylene, hydrogen peroxide
Cyanide	Acids, nitrates, nitrites
Flammable liquids	Ammonium nitrate, chromic and nitric acids, sodium and hydrogen peroxides, halogens
Fluorine	Isolate from everything; only Pb and Ni resist prolonged exposure
Glycerine	Chromic acid, permanganates
Hydrocarbon liquids and gases	Fluorine, chlorine, bromine, chromic acid, sodium peroxide
Hydrochloric acid	Nitrates, chlorates, oxidizers, metals
Hydrocyanic acid	Nitric acid, alkalies
Hydrofluoric acid	Ammonia (aqueous or anhydrous)
Hydrogen peroxide	Copper, chromium, irons, other metals, metal salts, flammable liquids, aniline, nitromethane
Hydrogen sulfide	Nitric acid, oxidizers
Iodine	Acetylene, ammonia, hydrogen
Lithium metal	Water, carbon dioxide, chlorinated hydrocarbons, halogens
Magnesium metal	Water, carbon dioxide, chlorinated hydrocarbons, halogens
Mercury	Acetylene, fulminic acid, ammonia
Nitric acid (concentrated)	Hydrocyanic acid, fulminates, chlorates, picrates, turpentine, carbide, metal powders, acetic acid, aniline, chromic acid, hydrogen sulfide, flammable liquids and gases
Oxalic acid	Silver, mercury
Oxygen (liquid or gas)	Oils, grease, hydrogen, flammable liquids, solids or gases
Perchloric acid	Acetic anhydride, bismuth, alcohols, flammables, dehydrating agents
Phosphorous (white)	Air, oxygen, oxidizers
Potassium chlorate	Acids, phosphorous, sulfites, metal powders, sulfur
Potassium metal	Water, carbon dioxide, chlorinated hydrocarbons, halogens
Potassium permanganate	Glycerine, ethylene glycol, benzaldehyde, sulfuric acid, alcohols, ether, flammable gases
Sodium chlorate	Acids, ammonium salts, metal powders, sulfur
Sodium chlorite	Acids, sulfur, combustibles
Sodium metal	Water, carbon dioxide, carbon tetrachloride
Sodium nitrate	Ammonium nitrate and other salts
Sodium peroxide	Alcohols, methyl acetate, acetic acid, acetic anhydride, benzaldehyde, carbon disulfide, glycerine, ethylene glycol, ether, furfural, glycerol
Sulfur	Sulfides, nitrates and oxidizers
Sulfuric acid	Water, light metals (Li, Na, K), sulfides, nitrates, nitrites, fluorides, bromides, iodides, fulminates, metal powders, carbides, picrates, chlorates, perchlorates, permanganates
Zinc powder	Acids, NaOH, KOH, moisture

The table above lists specific chemicals known to be reactive or otherwise incompatible. This information is intended to supplement, but not substitute for, reading the MSDS. A much more complete listing of incompatible and reactive substances can be found in [70]. Sources [70, 110, 175, 224].

Waste Effluent Disposal

Waste effluent disposal can be a significant contributor to overall process cost. The costs of proper disposal of hazardous materials can often outweigh the initial cost of the materials themselves. The impact will vary depending on whether the ultimate manufacturing site operates an in-house waste treatment plant or whether off-site disposal is required. The economic implications are not limited to mere disposal; there are also management, record-keeping and energy costs associated with minimizing worker exposure and the environmental impact of hazardous wastes.

Regulations – The Resource Conservation and Recovery Act (RCRA) and the Toxic Substances Control Act (TSCA) passed by Congress empower the EPA to establish regulations to ensure the disposal of hazardous industrial waste in a manner that protects the environment and human health. These regulations, along with local requirements, can be complex, and an in-depth knowledge of the correct policies and procedures is critical to ensure compliance with all laws and restrictions. The underlying philosophy behind EPA guidelines calls for a “cradle to grave” approach, wherein the company or organization generating the waste has full legal responsibility for it from the moment it is produced until it is safely disposed. In other words, responsibility for safe disposal is not transferred to the waste disposal contractor on pickup. It remains that of the generator, so choose waste disposal companies carefully to ensure their reliability.

The DOT also develops regulations for the labeling and transportation of hazardous materials, including hazardous wastes. The heart of the DOT regulations is the Hazardous Materials Table found in 49 CFR Part 172.101. This table provides important information, including proper shipping names, which must be observed. Additional requirements may include wastewater discharge standards established by the Clean Water Act, local sewer discharge rules, state water quality standards and maximum storage limits for various kinds of wastes.

Generator Classification – Waste generators are classified according to the amount of waste they produce. Regulations become more stringent the larger the size. Facilities that produce less than 100 kg per month of hazardous waste (or 1 kg/month of acutely toxic waste), while still bound to follow safe disposal practices, are exempt from many of the further requirements imposed on larger generators. Beyond that, a further distinction is made between “Small Quantity Generators” (SQG), producing less than 1000 kg of waste per month and “Large Quantity Generators” (LQG) producing more than 1000 kg per month.

SQGs and LQGs are required to apply for a unique EPA ID number, are subject to waste storage limitations, and must issue a waste manifest with each shipment. This manifest becomes a permanent record of the disposition. SQG’s are required to provide basic operator training and have a basic spill response plan, whereas LQG’s are required to institute a full training program and have a complete written emergency response plan.

Waste minimization – Legal requirements and the long-term liability potential are only two of the many good reasons to find ways to minimize the amount of waste generated. Look for and try to adopt waste minimization practices which might include:

- Establish a chemical inventory system to track incoming chemicals and help avoid unnecessary ordering.
- Avoid ordering large excesses of chemicals and solvents – consider the hidden costs of disposal of unused materials.
- Substitute less hazardous compounds where possible; use water for a solvent when possible.
- Optimize reaction and isolation yields and try to minimize formation of by-products and impurities.
- Run development experiments at smaller scale (use of small automated reactors).
- Make waste stream and disposal analysis part of route selection; include scrub liquors and cleaning solutions.
- Try to select solvents that are easily recycled and establish a recycle program.
- Try to develop more solvent-efficient equipment cleaning procedures.
- Participate in a surplus chemical exchange program.
- Concentrate waste streams or convert hazardous wastes to less harmful forms in-house.

Segregation and Storage – For off-site disposal, the principle types of liquid waste to be dealt with are non-halogenated flammable solvents, halogenated (nonflammable) solvents, aqueous waste saturated with non-halogenated solvents and

aqueous waste saturated with halogenated solvents. Designated containers for other waste types are commonly used. For example, acetonitrile is usually separated simply because of its high caloric content and lower incineration cost. Cyanide or mercury-containing wastes, among others, may have to be segregated as well. Various solid waste types are also to be segregated. Determine the exact requirements from your waste-disposal contractor.

Waste streams should be segregated at the point of generation. Clearly labeled containers for each type of waste should be set up in a designated waste storage area with adequate ventilation and grounding supplied. Depending on the size, safety cans or drums may be used. Safety cans are OSHA designated containers with a leak-tight spring-activated cover designed to relieve pressure in the event of a fire. These are generally 5-gallons or smaller. They should be fitted with flame arrestors, a metal screen in the pouring spout that helps prevent ignition and acts to squelch a flash-fire within the container by limiting the amount of oxygen available. In plastic safety cans, the metal arrestor is the only grounding point for the can. Never use a can for flammables without the flame arrestor in place. For bulk storage, steel drums are acceptable for organics and flammables. Aqueous-containing streams should be stored in poly-lined drums to prevent corrosion. In order to comply with storage-time restrictions, in addition to the normal labeling for identification, drums and other bulk containers must be labeled with the date on which the drum was put into service. Waste segregation can be considered a cost saving approach as well, because of the volume reduction that will result from keeping hazardous waste streams separate from nonhazardous waste streams.

Poly-lined drums are necessary for storing strongly acidic or basic waste (pH less than 2 or greater than 12.5) which can corrode many metals. However, many other substances can soften or weaken common liner materials and so it is important to understand the liner compatibility and its suitability for waste storage. Drum liners often used for shipping raw materials may not be adequate for hazardous waste storage. Detailed information should be available from the drum manufacturer.

Record Keeping – Establish a waste disposal document retention system and enforce its use. Generators are required by the EPA to keep shipping manifests and other waste generation and disposal records forever. Manifests should accompany all waste shipments. Use your state's form or the universal form in 49 CFR 262 Appendix. In-house environmental audits are encouraged as a way of demonstrating due diligence in terms of meeting government regulations.

Disposal Safety – Never add waste to a waste container without knowing the container's contents and understanding any possible effects of chemical interaction (see the list of incompatible chemicals on page 9-9). Acidic and basic aqueous wastes should be neutralized to between pH 4 and 10 prior to disposal. Always use adequate personal protective equipment, including a full face shield and heavy-duty gloves when transferring waste. Ensure adequate ventilation or respiratory protection. Leave room for expansion due to temperature changes.

Common Waste Treatment Processes – A number of standard waste treatment procedures are used throughout the industry. Some of these are listed below. For more information on the destruction and neutralization of various types of chemicals and wastes, see references [151, 164].

- Neutralization for basic or acidic wastes; may require several tanks for neutralization and final adjustment
- Oil/water separation or decantation to remove bulk organics for incineration or reprocessing
- Clarification – starting with coagulants or flocculents such as aluminum sulfate, sodium aluminate or ferric sulfate followed by filtration, sedimentation or centrifugation
- Chemical precipitation, oxidation or reduction
- Biological waste water treatment – aerobic and anaerobic processes, suitable for a surprising number of organic compounds and heavy metals
- Softening and ion exchange for final treatment, oxygen scavengers such as hydrazine or hydroquinone
- Activated carbon adsorption – a comprehensive list of organic compounds that can be adsorbed by activated carbon is provided in [151]. Carbon regeneration methods include steam, solvent cleaning or dry heat.
- Air stripping – to remove volatile organics from waste water, usually using venturi-type strippers
- Incineration – using suitable particulate and gas scrubbers to meet strict emission standards

Eye and Face Protection

All eye protection used in chemical plants and laboratories must bear the “Z87” mark indicating compliance with the stringent ANSI Z87.1 standard for occupational safety eyewear. Prescription street glasses generally do not comply to this standard. Normal impact-resistant safety glasses with side shields should be the minimum requirement for everyone, including guests, entering a location where chemicals are handled. However, these do not provide adequate protection against splashes, and therefore anyone actually involved in dispensing or handling chemicals should wear approved splash goggles. These also offer good protection from particulates and impact from flying objects. When handling particularly dangerous substances, concentrated acids for example, a full face shield should be worn, but always as a secondary layer of protection over goggles or safety glasses. Face shields should be large enough to protect ears and neck.

Always ensure that your eye protection fits properly and does not interfere with other equipment you may be using such as a respirator. Safety goggles should fit tight to your face. Never use eye protection that is badly scratched or damaged. Clean your equipment after chemical exposure.

The use of contact lenses has been a controversial subject for laboratory and industrial workers. It is now widely believed that wearing contact lenses will not increase the likelihood or severity of injury if the eyes are splashed. However, contacts offer no eye protection – so additional eye protection must always be worn.

Hearing Protection

Hearing loss caused by exposure to loud noise is irreversible. The safety limit set by OSHA is a noise level of no greater than 90 dB over a time-weighted average of 8 hours. For comparison, some typical sound level values are given below along with their effects. Note that the decibel scale is actually logarithmic, derived from actual sound energy intensity.

Understand the proper use of ear plugs and earmuff type coverings. Use such devices with caution, realizing that they can interfere with communication, and that careless insertion of earplugs could introduce contaminants into the ear. Headphones may be superior on both counts. Hearing protection devices are labeled with a noise-reduction rating (NRR) according to EPA requirements. These are determined in ideal laboratory settings, and so the real world sound reduction protection that they provide may actually be much less.

Comparison of Noise Levels

Loudness, Decibels	Sound Intensity, watts/m ²	Relative Noise Level	Examples
130	10 ¹³	Deafening	Pain Threshold
120	10 ¹²		Jet Engine
110	10 ¹¹		High Speed Train Passing
100	10 ¹⁰	Noisy	100 HP TEFC Electric Motor
90	10 ⁹		Noisy Street
80	10 ⁸		Home Shop Tools
70	10 ⁷	Average	Noisy Office, Restaurant
60	10 ⁶		Hair Dryer
50	10 ⁵	Quiet	Window Fan
40	10 ⁴		Museum, Library, Quiet Restaurant
30	10 ³	Very Quiet	Whisper at 5 ft.
20	10 ²		Very quiet residence
10	10	Threshold of Audibility	Sound proof room
0	1		Inaudible

Sources: [49, 142]

Hand Protection

Always protect your hands when working with chemicals. Never deliberately touch chemicals or solvents with your bare hands. Never use a solvent to wash, since it may speed skin adsorption. Be familiar with the safety characteristics of the substances you are using in case special precautions apply (water reactivity, etc). Always read the MSDS.

Chemical protective gloves are available in many materials, thicknesses and strengths. No one glove is suitable for all situations. Before beginning work, ensure that you are using the proper gloves for the purpose. Chemical protection from gloves is dependent on both degradation of the glove material itself as well as permeation rate of the substance through the glove. If no single glove is rated for protection against all of the chemicals you will be using at a given time, select the glove that protects against the most dangerous substance. Wear different gloves in layers if necessary to protect against multiple hazards, or to increase mechanical strength. Use sleeve coverings for additional protection.

Inspect gloves before use to ensure that they are not cut or damaged in any way and stop using them immediately if they become punctured or damaged. Wash your hands and get a new pair. Never reuse disposable gloves. Decontaminate reusable gloves after use. Wash your hands after removing gloves in case any material was absorbed through the gloves or you contaminated your hands when removing them.

Glove Selection Guide

The chart below lists some of the more common glove types and indicates their compatibility with various materials. For more complete information, consult the MSDS's for the materials you are handling. Glove manufacturers and material suppliers are also an excellent source of compatibility information. If in doubt about a critical application, gloves can be tested by immersing a small sample in the test solution for a half-hour or so and looking for signs of swelling, discoloration or changes in weight.

The data in the following table are adapted from a number of sources, some of which were occasionally contradictory. Use it as a general guide only. Note that glove thickness and manufacturing method will have a significant impact on rates of permeation, ability to resist degradation and overall suitability for a particular use. The best information will come from the manufacturer of the particular gloves in question. Sources [33, 63, 99, 219].

Glove Selection Guide

Glove Material	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton
Substance								
Acetaldehyde	A	B	C	D	C	D	D	D
Acetamide	D	D	C	B	B	-	-	-
Acetates	-	-	D	D	A	-	D	-
Acetic Acid	A	A	A	B	A	D	B	A
Acetic Anhydride	C	C	B	C	D	-	D	-
Acetone	A	D	C	D	B	C	D	D
Acetonitrile	A	C	B	D	-	A	C	D
Acetyl Chloride	B	B	D	D	D	D	D	D
Acrylonitrile	A	C	C	D	A	D	D	D
Allyl Alcohol	A	D	B	A	-	-	-	-
Amines	C	C	B	D	C	D	B	-
Ammonia gas	A	C	B	B	B	-	C	A
Ammonium Hydroxide	A	C	A	B	A	D	C	A
Amyl Acetate	C	D	C	C	C	B	D	D
Amyl Alcohol	A	A	A	A	B	B	C	A
Aniline	A	C	C	D	C	C	C	A
Antimony Trichloride	-	-	-	B	B	-	A	-
Aqua Regia	B	B	A	B	B	D	D	A
Aromatic Hydrocarbons	D	D	D	D	C	-	-	-
Benzaldehyde	A	C	D	D	A	B	D	C
Glove Material	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton
Substance								
Benzene	D	D	D	D	D	A	D	A
Benzoic Acid	D	D	D	D	A	-	B	-
Benzyl Alcohol	A	A	B	D	-	-	B	A
Benzyl Ether	-	-	D	D	-	-	-	-
Bleach solutions	D	D	B	D	-	-	B	-
Bromine, anhyd. liquid	D	D	D	D	D	-	D	-
Bromine dry gas	D	D	D	D	D	-	D	-
Bromobenzene	D	D	D	D	-	A	D	A
Bromotoluene	-	-	-	D	-	-	-	-
Butadiene	A	D	C	D	D	-	D	A
Butane	D	D	B	A	C	-	C	-
Butanol	A	A	A	A	B	C	B	A
Butyl Acetate	C	D	D	C	B	B	D	D
Butyl Ether	D	D	B	A	-	-	A	-
Butylamine	B	C	D	C	C	D	C	D
Butylcarbitol	A	A	A	A	-	-	A	A
Butylcellosolve	A	A	A	A	-	-	A	A
Butyraldehyde	-	-	D	D	-	-	-	-
Butyric Acid	A	D	D	C	D	-	D	A
Carbitol	A	A	A	B	-	-	-	-

Glove Selection Guide (continued)

Glove Material	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton	Glove Material	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton
Substance									Substance								
Carbon Disulfide	D	D	D	C	C	A	D	A	Ethyl Chloride	B	B	C	A	C	-	D	-
Carbon Tetrachloride	D	D	D	B	D	A	C	A	Ethyl Ether	D	D	C	C	D	A	D	D
Cellosolve	A	A	A	B	-	-	C	-	Ethylamine	-	-	C	D	-	-	-	-
Cellosolve Acetate	A	B	B	B	-	-	D	D	Ethylbenzene	D	D	D	C	-	-	C	-
Chloric Acid	-	-	D	-	-	-	-	-	Ethylene Bromide	C	C	C	D	D	-	D	-
Chlorine gas	B	B	D	D	D	-	B	A	Ethylene Chloride	D	D	D	D	D	-	-	-
Chlorine, anhydr. liquid	D	D	D	D	D	-	B	B	Ethylene Chlorohydrin	C	C	-	D	D	-	D	-
Chloroacetic Acid	B	D	B	D	B	D	D	A	Ethylene Diamine	D	D	A	B	A	-	-	-
Chlorobenzene	D	D	D	D	C	A	D	A	Ethylene Dichloride	C	C	D	D	D	A	D	-
Chlorobromomethane	D	D	-	D	A	-	-	-	Ethylene Glycol	A	A	A	A	A	B	A	A
Chloroform	D	D	D	D	C	A	D	A	Ethylene Glycol Butyl Ether	A	A	A	A	A	-	A	A
Chlorosulfonic Acid	D	D	D	D	D	-	C	-	Ethylene Glycol Hexyl Ether	A	A	A	A	-	-	A	A
Chlorotoluene	D	D	D	D	A	-	-	-	Ethylene Glycol Methyl Ether	A	A	A	A	-	-	A	A
Chromic Acid 50%	A	A	A	A	A	D	A	A	Ethylene Oxide	B	D	D	D	A	-	D	D
Cresol (o-, m-, p-)	A	B	A	D	C	-	A	A	Ethylene trichloride	-	-	D	D	-	-	-	-
Cyclohexane	D	D	B	A	B	D	C	A	Fatty Acids	B	B	B	B	D	-	B	-
Cyclohexanol	A	A	A	A	-	B	A	A	Flourine gas	C	C	-	D	D	-	D	-
Cyclohexanone	A	D	C	D	D	A	D	D	Flourine liquid	-	-	C	D	-	-	-	-
Cyclopentane	D	D	A	B	-	-	-	-	Fluoboric Acid	A	A	A	A	A	-	B	-
Decane	-	-	D	B	-	-	-	-	Fluosilicic Acid	-	-	A	A	A	-	B	-
Diacetone alcohol	A	A	A	B	A	-	B	B	Fluosilicic Acid	-	-	A	B	-	-	-	-
Dibutylamine	-	-	-	C	-	-	-	-	Formaldehyde, 37%	A	A	A	A	B	C	A	A
Dichlorobenzene, 1,2-	D	D	D	D	D	-	D	A	Formamide	-	-	A	A	-	-	-	-
Dichloroethane, 1,2-	C	D	D	D	C	-	D	A	Formic Acid, 90%	A	A	A	B	D	A	A	C
Dichloromethane	C	C	D	D	D	A	D	C	Freon 113	A	B	A	A	A	C	B	A
Diesel Fuel	D	D	B	A	C	-	B	A	Fuel Oils	D	D	B	A	B	-	D	-
Diethanolamine	A	A	A	B	-	-	A	A	Furan	D	D	D	D	D	C	A	D
Diethyl Ether	D	D	D	D	-	-	-	-	Furfural	A	A	B	D	D	C	B	B
Diethylamine	C	D	C	D	D	D	D	D	Gasoline	D	D	C	B	A	B	B	A
Diethylene Glycol	A	A	A	A	B	C	C	A	Glutaraldehyde, 50%	A	A	A	A	-	-	B	A
Dimethyl Aniline	D	D	-	D	-	-	D	-	Glycerol	A	A	A	A	A	-	B	-
Dimethylformamide	C	C	D	D	A	-	D	-	Heptane	D	C	B	A	B	C	D	A
Dioxane	-	D	D	D	-	-	-	-	Hexane	D	D	B	A	D	A	C	A
Diethylene Gly. Butyl Ether	A	A	A	A	-	-	A	A	Hexanol, 1-	A	A	B	A	-	-	-	-
Diethylene Gly. Diethyl Ether	A	A	B	B	-	-	A	A	Hexylene Glycol	-	-	A	A	-	-	-	-
Diethylene Gly. Ethyl Ether	A	A	B	B	-	-	A	A	Hydraulic Fluids	D	D	B	D	C	-	A	-
Diethylene Gly. Hexyl Ether	B	B	A	A	-	-	A	A	Hydraulic Oil (Synthetic)	D	D	-	D	A	-	-	-
Diethylene Gly. Methyl Ether	A	A	B	A	-	-	A	A	Hydrobromic Acid	A	A	C	A	B	D	A	-
Diisobutyl Ketone	A	B	A	A	-	B	A	A	Hydrochloric Acid 37%	A	B	B	A	B	D	B	A
Dimethylacetamide, N,N-	A	C	C	D	D	D	D	D	Hydrofluoric Acid 48%	A	B	A	B	A	D	B	A
Dimethylformamide	A	A	B	D	-	D	D	D	Hydrofluoroether HTF's	A	A	A	A	-	-	-	-
Dimethyl Sulfoxide	A	A	A	A	-	D	D	C	Hydrogen Chloride gas	A	A	A	B	A	-	C	A
Dioxane, 1,4-	B	B	C	D	D	C	D	D	Hydrofluosilicic Acid 100%	A	A	A	B	B	-	C	-
Dipropylene G. Butyl Ether	A	A	A	A	-	-	A	A	Hydrogen Peroxide (30%)	A	A	A	A	C	D	A	A
Dynalene HTF's	B	B	B	-	-	-	-	A	Hydrogen Peroxide (50%)	B	B	B	B	C	D	B	A
Ethanol	A	A	A	A	B	D	B	A	Hydrogen Sulfide (aq)	C	C	B	D	A	-	B	-
Ethanolamine	A	A	A	A	-	C	A	A	Hydroxyacetic Acid 70%	-	-	A	A	A	-	-	-
Ethoxyethanol, 2-	A	C	B	D	-	D	D	D	Hypochlorous Acid 25%	-	-	D	D	-	-	-	-
Ethyl Acetate	B	C	C	C	A	C	D	D	Iodoform	B	B	A	D	-	-	C	-
Ethyl Benzene	D	D	D	D	C	D	D	A	Isoamyl Acetate	C	D	D	D	D	-	D	D
Ethyl Bromide	A	A	B	D	-	-	-	-	Isocetane	D	D	A	A	B	A	C	A
Ethyl Cellosolve	-	-	C	C	-	-	-	-	Isoamyl Alcohol	A	A	A	A	-	-	B	A

PVAL = polyvinyl alcohol PVC = polyvinyl chloride

continued next page...

Legend:

A - Safest choice

B - Acceptable choice, change if exposed

C - Poor choice, protects against splashes only, change quickly

D - Very poor choice, offers little or no protection

- no data available

Glove Selection Guide (concluded)

Substance	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton	Substance	Butyl Rubber	Natural Rubber	Neoprene	Nitrile	Polyethylene	PVAL	PVC	Viton
Isobutanol	A	A	A	A	-	C	B	A	Oil (mineral)	B	B	B	A	-	-	-	-
Isobutyl Acetate	D	D	D	D	-	-	-	-	Oils (silicone)	A	A	D	A	B	-	B	-
Isobutyl Chloride	-	-	D	D	-	-	-	-	Oils (vegetable)	A	A	A	A	A	-	A	A
Isooctane	C	B	A	A	-	-	B	A	Oleic Acid	C	C	A	A	C	B	C	-
Isopropanol	A	A	A	A	D	D	B	A	Oxalic Acid (aq)	A	A	A	A	A	C	A	-
Isopropyl Acetate	D	D	D	D	B	-	D	-	Ozone	D	D	B	D	C	-	A	-
Isopropyl Ether	D	D	D	C	B	-	D	-	Paraffins	-	-	-	A	B	-	D	-
Isotane	-	-	D	A	-	-	-	-	Paraformaldehyde	-	-	B	B	-	-	-	-
Kerosine	D	D	A	A	C	B	A	A	Pentane, n-	D	D	B	A	D	B	B	A
Ketones	B	B	D	D	C	-	D	-	Perchloric Acid	C	C	A	A	B	D	A	-
Lactic Acid 85%	A	A	A	A	A	B	A	A	Perchloroethylene	D	D	D	B	D	A	D	A
Laquer Thinner	D	D	D	D	A	-	D	C	Petroleum Ether (naptha)	D	D	D	B	A	A	C	A
Lacquers	D	D	D	D	A	-	D	-	Phenol	A	C	B	D	D	C	B	A
Limonene-D	D	D	C	B	-	A	B	A	Phosphoric Acid 85%	A	A	A	A	B	D	A	A
Linoleic Acid	D	D	D	B	A	-	-	-	Piperidine	D	D	D	D	-	-	D	-
Linseed Oil	D	D	D	A	-	-	B	-	Polyvinyl Acetate	-	-	C	-	-	-	-	-
Mercury	A	A	A	A	A	-	A	-	Potassium Hydroxide, 50%	A	A	A	A	-	D	A	A
Methanol	A	A	A	A	A	D	B	A	Propanol, 1-	A	B	A	A	-	C	A	A
Methanolamine	A	A	B	A	-	D	A	-	Propyl Acetate	C	D	C	C	-	B	D	D
Methyl Acetate	B	C	D	D	B	-	D	D	Propylene	D	D	-	D	-	-	A	-
Methyl Acetone	A	A	D	D	-	-	A	-	Propylene Glycol	-	-	C	A	B	-	-	-
Methyl Acrylate	D	D	B	D	-	C	D	D	Propylene Gly. Butyl Ether	A	A	A	A	-	-	A	A
Methyl Amine	A	B	A	B	A	-	C	-	Propylene Gly. Methyl Ether	A	A	A	C	-	-	C	B
Methyl Bromide	D	D	D	C	C	-	D	-	Propylene Gly. Propyl Ether	A	A	B	C	-	-	B	A
Methyl Butyl Ketone	D	D	D	D	-	-	-	-	Propylene Oxide	D	B	D	D	D	C	D	D
Methyl Carbitol	A	A	A	A	A	A	-	-	Pyridine	C	C	D	D	B	B	D	C
Methyl Cellosolve	A	B	A	C	-	B	C	D	Pyrrrole	-	-	D	D	-	-	-	-
Methyl Chloride, gas	A	B	B	B	C	-	A	A	Sea Water	A	A	B	A	A	-	B	-
Methyl Dichloride	D	D	D	D	-	-	D	C	Sodium Hydroxide 50%	A	A	A	A	A	D	A	A
Methyl Ethyl Ketone	B	D	C	D	D	C	D	D	Sodium Hypochlorite, 5%	A	A	A	A	-	-	A	A
Methyl Formate	-	-	B	D	-	-	-	-	Sulfur Dioxide gas (wet)	D	D	B	D	B	-	B	-
Methyl Iodide	D	D	D	D	D	B	D	A	Sulfur Trioxide (wet)	D	C	C	C	-	-	A	-
Methyl Isobutyl Ketone	B	D	D	C	C	B	D	D	Sulfuric Acid, 47%	A	A	A	A	A	D	A	A
Methyl Isopropyl Ketone	D	D	D	D	D	-	-	-	Sulfuric Acid, 97%	A	A	B	D	A	D	D	A
Methyl Methacrylate	C	C	D	D	D	B	D	D	Tetrachloroethane	D	D	D	D	-	A	D	A
Methylene Bromide	D	D	D	D	-	B	D	-	Tetrachloroethylene	D	D	D	C	B	A	D	A
Methylene Chloride	C	D	D	D	D	B	D	-	Tetraethylene Glycol	-	-	-	A	-	-	-	-
Methyl Propyl Ketone	-	-	D	D	-	-	-	-	Tetrahydrofuran	D	D	D	D	C	C	D	D
Methyl Pyrrolidone N-	A	A	B	D	-	D	D	D	Therminol D-12 HTF	-	-	C	B	C	-	-	-
Methyl t-Butyl Ether	D	D	D	B	D	B	D	D	Thionyl Chloride	-	-	D	D	-	-	-	-
Mineral Spirits	D	D	A	A	B	A	B	A	Toluene	D	D	D	C	C	B	D	A
Monoethanolamine	A	A	A	A	C	C	A	-	Trichloroacetic Acid 90%	D	D	D	-	A	-	A	-
Motor oil	-	-	B	A	C	-	D	-	Trichloroethane, 1,1,1-	D	D	D	D	D	B	C	A
Naphtha	D	D	B	A	A	A	C	A	Trichloroethylene	D	D	D	D	D	A	D	A
Napthalene	D	D	D	D	C	-	D	-	Trichloropropane	D	D	A	D	-	-	D	-
Nitric acid, conc.	A	A	A	D	C	D	C	A	Triethanolamine	A	B	A	A	-	B	A	A
Nitrobenzene	A	C	C	D	C	B	D	A	Triethylamine	B	B	B	A	-	-	A	-
Nitromethane	A	B	B	D	A	B	B	D	Trisodium Phosphate	A	A	B	B	A	-	A	-
Octane	D	D	C	A	A	-	D	A	Turpentine	D	D	A	A	D	B	B	A
Octanol, 1-	A	A	A	A	-	B	B	A	Vinyl Acetate	D	D	B	D	A	-	D	-
Oils (animal)	D	D	C	A	-	-	-	-	Vinyl Chloride, gas	A	D	D	C	-	-	C	A
Oils (lubricating, petroleum)	B	C	A	A	C	-	A	-	Xylenes	D	D	D	D	B	A	D	A

PVAL = polyvinyl alcohol PVC = polyvinyl chloride

Legend:

A - Safest choice

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C - Poor choice, protects against splashes only, change quickly

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- no data available

Respiratory Protection

Respiratory protection is necessary under three conditions defined by OSHA: for particulates, for harmful gases and vapors, and in oxygen deficient atmospheres. A wide choice of protective devices is available and it is important to know that the equipment you are using is appropriate for the situation. A brief description of some is provided here.

Particulates – Fine particulate matter suspended in the air, many of which may be invisible, can be harmful in a number of ways if inhaled. Damage to the lungs, bronchi and other organs can result, and if the dust derives from a hazardous substance, the effects are compounded.

Most particulates can be removed using particulate respirators. These can range from simple dust masks to specially designed masks containing activated carbon for removing nuisance levels of organic vapors and acid and chemical mists. HEPA filter dust masks are a special type designed to trap extremely fine particulates. Never use a dust mask-type respirator where significant quantities of organic vapors exist or in an oxygen-deficient atmosphere. Doing so can create a false sense of security and do more harm than good. Do not reuse disposable masks, in case they have become contaminated on the inside.

Harmful gases and vapors – The next level of protection comes from respirators that use replaceable chemical cartridges or filters to remove chemical vapors from the air. Since there are so many types of cartridges available, it is important to know that you are using the right ones. The table at the bottom of this page lists some of the common respirator cartridge types. One widely used style is the half-mask respirator, but for a higher level of protection when handling chemicals, a full-face respirator is recommended.

All air-purifying respirators must be individually fitted to the user and tested to ensure the tightness of the seal. Many companies strictly forbid any employee from using a respirator without training, certification and being fitted for his or her own respirator. This is sound practice since improper use of a respirator, as already mentioned, will only increase the chances of injury. Note that beards, mustaches and the like can affect the fit of the respirator.

Oxygen deficient atmospheres – This condition exists when the concentration of O₂ is less than 19.5% (normal air contains 21%). Long term exposure to low oxygen concentrations can impair judgment and cause unconsciousness and death. When working in such environments (for example, inside a vessel) the worker must use a positive pressure, or pressure-demand supplied air respirator, continuous flow air hood, or a self-contained breathing apparatus (SCBA). The use of such devices requires certification based on very specific training, respiratory capacity tests, and individual equipment fit tests. These functions must be performed by a certified safety professional. Using such safety systems without certification is extremely dangerous and illegal in most situations.

Never use a supplied air system of any kind without ensuring for yourself that the system is working properly, that the correct gas mixture is being used (typically medical-grade air) and that it is properly connected. Never work alone when wearing such equipment.

Each time a respirator is used and before entering a hazardous environment, test the fit by covering the filters with your hands and trying to inhale and then exhale to ensure that there are no leaks around the face seal. Always clean the respirator after use according to manufacturer's instructions. Store it in a sealed bag in a clean dry place. Change cartridges frequently – exhausted cartridges are worse than no cartridges at all.

Chemical Cartridges for Air-Purifying Respirators

ANSI Standard Color Code	Contaminant	Examples (always refer to individual manufacturer's labeling)
White	Acid Gases only	chlorine, HCl, HF, sulfur dioxide
Black	Organic Vapors only	hydrocarbons, alcohols, ketones, etc.
Green	Ammonia Gas	ammonia gas
Yellow	Acid Gases and Organic Vapors	chlorine, HCl, HF and organic vapors
Purple	Aerosols	oil mists, asbestos dusts and some radionuclides
Orange	Mists and Fumes	mercury vapors and chlorine
Brown	Other	methylamine, formaldehyde

Source: [144]

10 Materials Selection

Contents

MATERIAL PROPERTIES

Properties of Common Elastomers	10-2
Properties of Common Fluoropolymers	10-3
Properties of Common Plastics	10-4
U.S. Plastics Recycling Symbols	10-4
Properties of Common Metals	10-5

CORROSION

Corrosion	10-6
Relative Corrosion Resistance of Various Materials	10-6
Corrosion Units Conversion Factors	10-7
Galvanic Series of Metals	10-7

GLASS

Properties and Corrosion of Glass	10-8
Effect of pH on Glass Corrosion Rate	10-8
Substances Known to Attack Borosilicate Glass	10-8
Corrosion of Borosilicate Glass by Acids	10-9
Corrosion of Borosilicate Glass by Bases	10-9

MATERIAL COMPATIBILITY

Material Compatibility Table	10-10
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METAL ATTACK

Substances Known to Attack 316 Stainless Steel	10-28
Substances Known to Attack Hastelloy C-276	10-28
Substances Known to Attack Tantalum Metal	10-28

Properties of Common Elastomers

Type	Trade Names	Recommended Temp Range °C	Characteristics/Identifiers
ACM (polyacrylate) or AR (acrylic rubber)	Hycar, Thiacyrl	-45 to 170	Transparent, heat and oil resistant, but not for use with steam or organic chemicals such as ketones and esters.
Chlorobutyl rubber	Norprene	-60 to 135	Good heat, chemical, ozone, UV resistance.
CO, ECO (epichlorohydrin rubber)	Hydrin	-40 to 125	Resistant to diffusion of gases and to solvents, oil and ozone, but not strong mineral acids or chlorine.
CR (polychloroprene rubber, or chlorinated butadiene polymer)	Neoprene, Duprene	-35 to 140	Strong, outstanding oil and oxidation resistance, poor aromatic HC resistance, mechanically similar to NR. Gaskets may be color coded with one yellow mark.
CSM (chlorosulfonated polyethylene elastomer)	Hypalon	-60 to 120	Good heat, chemical, ozone and solvent resistance.
EPM, EPR, EPDM (ethylene propylene and ethylene propylenediene rubber)	Nordel	-60 to 145	Poor oil and hydrocarbon resistance, outstanding resistance to ozone and weathering. Black or white in color, gaskets may be marked with one white stripe and two yellow dots, or one green dot, one blue dot or three green dots.
IIR (isobutylene isoprene copolymer)	Butyl Rubber	-60 to 140	High elasticity, good dielectric, exceptional acid/base resistance (except conc. HNO ₃ and H ₂ SO ₄).
Latex (synthetic cis-1,4-polyisoprene)	Isolene, Nipol IR	-50 to 100	Deteriorates in sunlight, attacked by hydrocarbons, halogens and conc. H ₂ SO ₄
NBR (nitrile or butadiene-acrylonitrile copolymer)	Buna-N	-60 to 130	Good abrasion and chemical (especially oil and fuel) resistance, not USP cytotoxicity certified. Black or white in color, gaskets may have one red or pink dot.
NR (natural rubber)	Denflex, others	-72 to 80	Flexible, deteriorates in sunlight, attacked by oxidizers, oils, benzene and ketones.
Polyphenylene oxide	Noryl		Good dielectric, heat resistant, often glass-reinforced.
Polysulfide or alkyl polysulfide	Thiokol ST	-45 to 100	Flexible, good chemical resistance.
Polyurethane	Vibrathane, Vulcollan	-30 to 100	Abrasion and organic solvent resistance, brittle at low temps.
PVC (with elastomers)	Tygon	-30 to 70	Transparent, flexible, poor solvent resistance, not autoclavable.
SBR (styrene butadiene rubber)	Buna-S	-80 to 200	High elasticity, abrasion and crack resistance, low resistance to organic chemicals, deteriorates in sunlight.
Silicone (polydimethylsiloxane rubber)	Norsil, Permaflex	-100 to 260	Extremely flexible over wide temperature range, low porosity, virtually free of extractables, no taste or odor. Autoclavable. Swells in non-polar solvents. Translucent, gaskets may have one pink dot.
TPE (thermoplastic polyester elastomer)	Hytrel	-72 to 120	Good heat stability, low temperature flexibility, cream or tan in color.

Use the characteristics and temperature limits in this and the following tables as a general guide only. Many proprietary polymers and elastomers are blended or reinforced with other materials to improve wear characteristics and service ratings. For critical applications, check with the vendor or manufacturer. For more detailed chemical compatibility information, see the table beginning on page 10-10. Sources [18, 37, 71, 105, 142, 256].

Properties of Common Fluoropolymers

Type	Trade Names	Recommended Temp Range °C	Characteristics/Identifiers
ECTFE (ethylene-chlorotrifluoroethylene copolymer)	Halar	-100 to 150	High heat, flame and abrasion resistance, low creep, high impact strength, good dielectric. Subject to attack by amines, esters and ketones, especially at high temperatures.
ETFE (ethylene-tetrafluoroethylene copolymer)	Tefzel, Hostafion ET	-100 to 150	Translucent, good mechanical properties, excellent abrasion resistance, chemically inert, excellent dielectric.
FEP (fluorinated ethylene-propylene copolymer)	FEP resins	-200 to 200	Translucent, highly flexible, chemically inert, good dielectric, less thermally stable and more easily molded than teflon, used for pump/pipe linings.
FMQ or FSR (fluorosilicone rubber)	Sylon FX	-70 to 215	Translucent, chemically inert.
MFA (monofluoroalkoxy copolymer)	Hyflon	to 230	Transparent, chemically inert.
PCTFE (polychlorotrifluoroethylene)	Kel-F, Hostafion, Fluorothene	-30C to 160	Transparent thermoplastic, machinable, low cold-flow, good dielectric. Similar chemical resistance to teflon, but subject to swelling by ketones, chlorinated and aromatic compounds.
PFA (perfluoroalkoxy copolymer)	Teflon PFA	-195 to 260	Thermoplastic, excellent stress and weather resistance, chemically inert. Often used for linings.
Propylene-tetrafluoroethylene copolymer	Aflas, Dyneon BRF	-20 to 230	Resists acids, bases, steam and petroleum-based solvents even at high temperature. Rel. high cost.
PTFE (polytetrafluoroethylene)	Teflon, Hostafion TF, Fluon, Aflon TFE	-195 to 260	Opaque thermoset, usually white in color, low friction, high electrical resistance, chemically inert, high creep, insoluble in all known solvents. Not resistant to fluorine gas at high temperatures, or to molten alkali. Not recommended as gasket material when high temperature fluctuations are anticipated as it lacks "elastic memory". PTFE-enveloped gaskets are preferred.
PVDF (polyvinylidene fluoride)	Kynar, Hylar	-40 to 150	High abrasion resistance, low creep, good mechanical strength, attacked by ketones/acetates and strong bases. Often carbon filled for better electrical grounding.
PVF (polyvinyl fluoride)	Tedlar	-70 to 110	Durable, chemically inert, high tear strength, soluble in polar solvents above 100°C.
TFE-PMVE (tetrafluoroethylene-perfluoromethylvinyl ether copolymer)	Kalrez	-20 to 320	Flexible, exceptional chemical resistance. Kalrez is a specific proprietary formulation that exhibits unexcelled chemical resistance.
VDF-HFP (vinylidene-hexafluoropropylene copolymer)	Viton, Fluorel	-30 to 260	Flexible, black or white in color. Should not be used for steam. Gaskets may have one white and one green dot or one white and one yellow dot.
Vinylidene fluoride-pentafluoropropylene linear copolymer	Technoflon SL	to 180	Flexible, chemically inert.

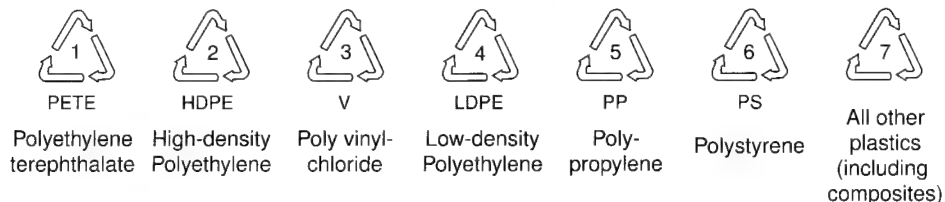
Commercial fluoropolymers are typically mixtures of four principle fluorolefin monomers – tetrafluoroethylene (TFE), vinyl fluoride (VF), vinylidene fluoride (VDF), and chlorotetrafluoroethylene (CTFE). These are often copolymerized with other polymers such as ethylene, propene, and hexafluoropropene. They generally exhibit excellent chemical resistance, high electrical resistance and are rated for use over a wide temperature range. To enhance their mechanical properties, such as wear resistance, they are often filled or reinforced with other materials, such as glass or bronze. The table above lists many of the common commercially available fluoropolymers along with some of their trade names and important properties. Sources [18, 37, 256, 105, 142].

Properties of Common Plastics

Type	Trade Names	Recommended Temp Range °C	Characteristics
ABS (acrylonitrile-butadiene-styrene copolymer)	Cyclocac	-40 to 80	Weakened by prolonged exposure to sun, poor resistance to aromatic and chlorinated solvents and oxidizing acids.
Acetal (polyacetal polyoxymethylene)	Lubetal, Delrin	to 120	High strength, very stiff, good abrasion and surface friction resistance, good machining properties, good organic solvent, but poor acid/base resistance. Should not be used on oxygen service.
Acrylic (mainly methyl methacrylate)	Plexiglass, Lucite	to 65	Highly transparent thermoplastic, rigid, machinable, good dielectric, low abrasion and scratch resistance, poor solvent but good acid/base resistance. Not autoclavable.
CPVC (chlorinated polyvinyl chloride)	Temprite	to 105	Thermoplastic, rigid, machinable, poor solvent resistance.
Epoxies	Epilite, Epikote	to 240	Thermosets, superior thermal and dimensional stability, excellent solvent resistance. Often fiber-reinforced.
Furan polymers	Furane, Quacorr	to 160	Thermosets, non-petroleum-based, good acid, alkali and solvent resistance.
Nylon (polyamides)	Zytel, Nylon, Novamide	0 to 140	Opaque, rigid, machinable, high tensile strength and abrasion resistance, can swell in aqueous environments. Not autoclav.
PEEK (Polyetheretherketone)	Xtrex	to 250	Excellent thermal stability, good chemical resistance.
Phenolic	Durez, Tufnol	to 250	Thermoset, superior heat and flame resistance, dimensionally stable.
Polycarbonate	Lexan	-130 to 150	Transparent, rigid, high impact resistance, machinable, good dielectric, high heat and flame resistance but poor solvent (aromatics, esters, ketones) and base resistance.
Polyester	Ampal, Palatal	to 200	Thermoplastic or thermoset types, tough, abrasion resistant, additives commonly used to improve chemical resistance.
Polyethylene	PE, Hostalen, Marlex	-100 to 90	Lowest-cost, most flexible thermoplastic, opaque. Low-density, high density and ultra-high-mol-wt. types available. Good solvent resistance, poor mechanical properties above 50°C.
Polyimides		to 250	Used for high temperature gears and bearings.
PPS (polyphenylene sulfide)	Ryton	to 250	Stable, temperature and chemical resistant, good dielectric
Polypropylene		0 to 130	Translucent, rigid, high strength, machinable, solvent resistant can be glass-filled for added strength.
Polystyrene	Dylene	20 to 90	Transparent, rigid, not solvent resistant, not autoclavable.
Polysulfone	Udel, Grafil	-100 to 150	Tough, rigid, subject to environmental stress cracking if not fiber reinforced. Swells in ketones, aromatic and chlorinated solvents.
Polyurethane		-270 to 120	Extremely tough, abrasion and tear-resistant, good oil and solvent resistance, highly flexible.
PVC (polyvinyl chloride), Type 1 (unplasticised)	Vinoflex, Vynaloy	-30 to 80	Thermoplastic, high fatigue strength, poor chlorinated solvent resistance, brittle below -30°C. Not autoclavable.

Sources: [18, 37, 105, 142, 256, 264]

U. S. Plastics Recycling Symbols



Properties of Common Metals

Metal	Density g/cm ³	Thermal Conductivity BTU/hr-ft-°F at ~100°C	Specific Heat Btu/ lb-°F at ambient	Characteristics
Aluminum	2.7	137	0.215	High strength/weight, easily fabricated but not easily soldered or welded, attacked by strong bases but resists acid attack because of protective surface oxidation. Usually alloyed with Zn or Mg to improve chemical resistance.
Brass (67% Cu, 33% Zn)	8.4	72.2	0.091	Easily soldered/brazed.
Copper	8.5	228	0.092	Relatively inexpensive with fair mechanical strength, easily fabricated and soldered. Good resistance to alkalis but not acids, oxidizes easily.
Gold	19.3	181	0.031	Excellent chemical resistance, soft.
Iron (cast)	7.5	26.6	0.100	Rusts and corrodes easily.
Iron (wrought)	7.7	31.8	0.110	Rusts and corrodes easily.
Lead	11.3	20.2	0.031	Excellent corrosion resistance except acetic / nitric acid.
Nickel	8.9	48.5	0.106	Weldable, resistant to strong bases, as hard as carbon steel.
Platinum	21.5	75.1	0.031	Excellent chemical resistance, high cost.
Silver	10.5	243	0.057	Expensive, low mechanical strength, but good chemical resistance to alkalis and organic acids.
Tantalum	16.6	28.8	0.034	Corrosion resistant properties similar to glass, attacked only by hot concentrated alkalis and hydrofluoric acid. High cost, poor fabricability, experiences accelerated atmospheric oxidation above 430°C. Often used in alloys.
Titanium	4.51	12.1	0.124	High strength, low weight, many alloys and crystal structures available.
Steels and Stainless Steels				
Carbon Steel	7.8	24.2	0.110	Strong, rusts easily, but acceptable for concentrated H ₂ SO ₄ .
Mild Steel	7.8	32.9	0.107	Rusts easily.
301 SS (18% Cr, 8% Ni, 0.15 C)	7.9	9.4	0.119	Good structural qualities for bins and containers.
302 SS (18% Cr, 9% Ni, 0.15 C)		9.4	0.119	Basic, general purpose austenitic type, good corrosion resistance.
304 SS (19% Cr, 10% Ni, 0.08 C)	7.9	9.4	0.114	Lower-carbon version of 302 (minimizes carbide precipitation during welding). Also as 304-L (0.03% C).
308 SS (20 % Cr, 10% Ni)	8.0	8.8	0.119	High heat and corrosion resistance.
314 SS (24% Cr, 20% Ni, 2% Si)	7.72	10.1	0.119	Resistant to oxidation in air up to 1000°C
316 SS (18% Cr, 12% Ni, 2-3% Mo)	8.0	9.4	0.119	Excellent corrosion and pitting resistance and high temperature strength. Available as 316-L for welded construction.
317 SS (18% Cr, 12% Ni, 3-4% Mo)	8.0	9.4	0.119	Highest aqueous corrosion resistance of all SS.
410 SS (12% Cr)	7.7	14.4	0.110	Lowest cost General purpose SS. Used where corrosion is not expected to be severe.
430 SS (16% Cr, 0.1% C)	7.7	15.1	0.110	Excellent heat and corrosion resistance to nitric acid and other oxidizers.
Superalloys				
Hastelloy B (63% Ni, 28% Mo, plus Fe, Co)	9.24	7.1	0.091	Excellent chemical and oxidation resistance at high temperatures. Very high strength.
Hastelloy C (56% Ni, 17% Mo, 16.5% Cr plus W, Fe)	8.94	5.7	0.101	Excellent resistance but not strong oxidizing acids or hot mineral acids in the presence of oxidizing cations (i.e. Fe III). High strength. First choice for reactors if glass unacceptable.
Inconel 600 (76% Ni, 16% Cr, 8% Fe plus Mn, Si, C)	8.42	6.9	0.103	Excellent resistance to oxidation, chloride ion and caustic at high temperatures.
Monel 400 (67% Ni, 33% Cu plus Mn, Si, C)	8.8	14.1	0.099	Good corrosion resistance (acids, bases, brines, HF).
Nichrome (67% Ni, 24% Fe, 16% Cr, 0.1% C)	8.4	8.1	0.103	High electrical resistivity, good corrosion resistance.

There are literally hundreds of industrially important alloys and composites with wide-ranging properties. A number of important nickel alloys are called superalloys because of their outstanding strength and oxidation resistance at high temperatures. Stainless steels are divided into several types: Ferritic (low C, high Cr to improve corrosion resistance), Austenitic (2nd major alloy is Ni giving better corrosion and temperature resistance), and Martensitic, (heat treated to varying degrees for various strengths). For more information on metal corrosion, see page 10-6. Sources [20, 30, 37, 105, 117, 139, 142, 256, 266]

Corrosion

Corrosion is a broad term used to describe the chemical attack and degradation of solid materials, primarily metals. There are a number of different types of metallic corrosion, caused by various chemical and mechanical phenomena including galvanic action, stress, wear, erosion and cavitation. This last category of corrosion is caused by liquid **cavitation** (for example in high speed pump rotors) where the rapid formation and collapse of minute vapor bubbles repeatedly hammer the surface. This can cause the surface to become brittle, flake off and become pitted, thereby making it more susceptible to chemical corrosion.

Stress corrosion can occur when metals are subjected to high tensile or cyclic stresses that can lead to metal fatigue. The weakened points are then more subject to chemical attack in corrosive environments which in turn can lead to cracking and mechanical failure. Similarly **fretting** corrosion occurs at points of sliding and friction between surfaces under load, again leading to increased susceptibility to chemical attack. This type of corrosion most severely affects metals that depend on a layer of surface oxidation for protection, e.g. aluminum. It can be controlled by keeping the surfaces lubricated or sealed, and minimizing movement or vibration. **Erosion** can occur if the surface is exposed to high velocity moving fluids, sometimes causing degradation in cases where it would not occur in the static fluid alone.

Another type of corrosion of great concern in the chemical processing industry is called **galvanic** corrosion. This is caused by electrochemical action in electrolytic environments (such as salt water) due to differences in oxidation potential between two dissimilar metals (or even different lots of the same metal) that are near or in contact with each other. A galvanic cell is formed in such a situation, with a current flowing from the metal with the higher oxidation potential (acting as the anode), to the lower (acting as the cathode). The cathode metal remains unchanged but the anode metal corrodes, and the degree of corrosion depends largely on the magnitude of the difference in potential between the metals. Often, the situation is made worse by the fact that crevices exist at points of contact between dissimilar metals, for example at threaded fittings, in which ions may build up and the attack becomes more severe. Reduced oxygen concentration at these points also contributes to the problem. This is referred to as **concentration cell** corrosion.

Corrosion at the anode is caused by the formation of stable salts and other complexes between the metal and process fluid. Inhibitors, such as some phosphates and silicates, are often added to process fluids to minimize galvanic corrosion with good success. Galvanic corrosion may also be prevented by using inert spacers to eliminate or minimize contact between the metal surfaces, minimizing the use of threaded connections, or using coatings or platings to protect the

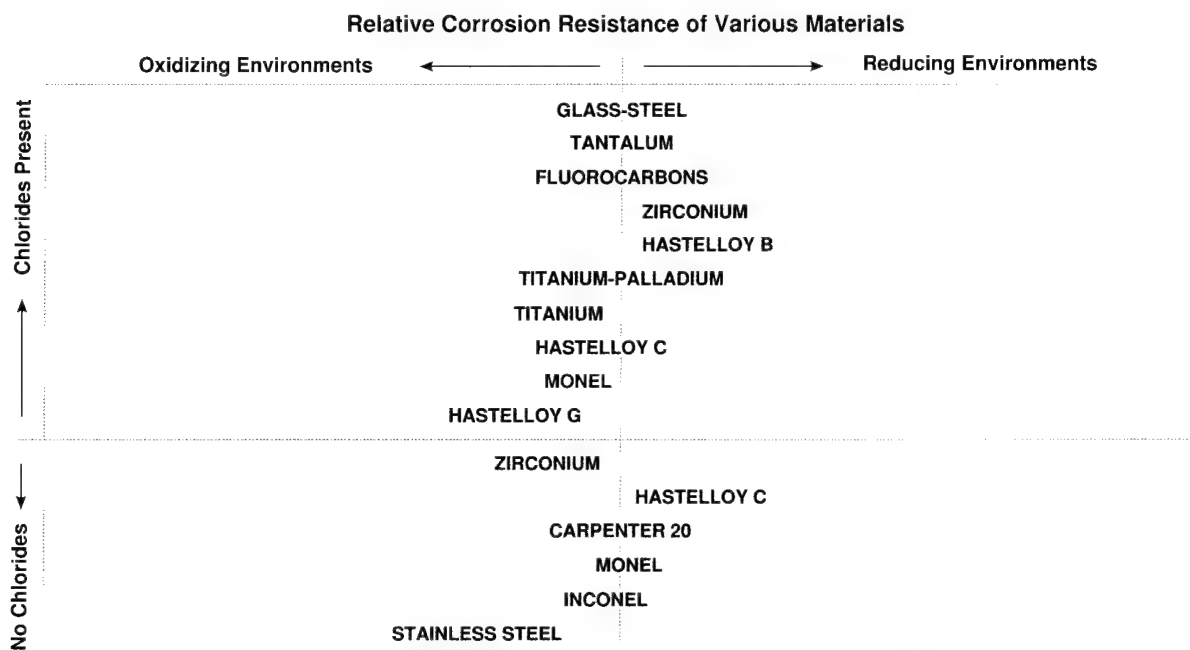


Chart courtesy of Pfaudler, Inc.

metals. However, when metal protective platings are used, such as chromium on steel, microscopic pores in the surface can promote subsurface corrosion that may go undetected. Glass-lined or PTFE-lined piping or vessels are another successful way to prevent corrosion. However, since these materials are more subject to breakage or mechanical failure, care must be taken to properly install and maintain such systems.

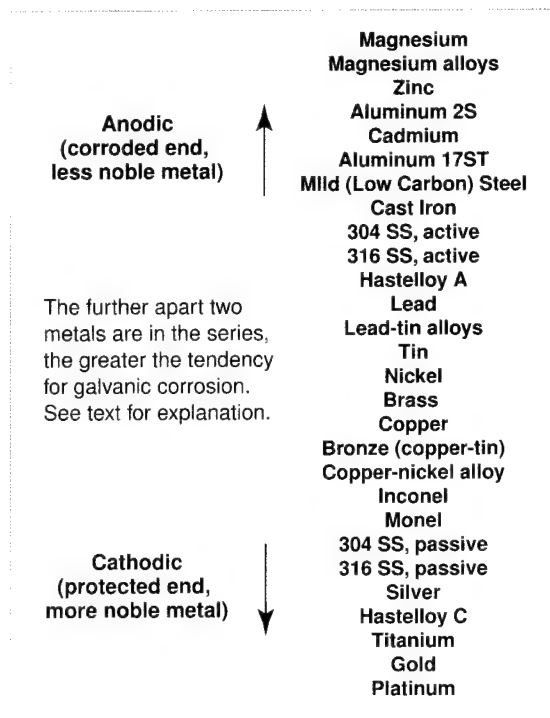
The best approach when dissimilar metals must be used is to select metals that are as close as possible in oxidation potential. The list of metals shown to the right is called the **galvanic series**. The closer two metals are to each other in the series, the smaller the difference in their oxidation potentials and therefore the lower the tendency toward galvanic corrosion. The farther apart the metals are in the series, the greater the galvanic tendency, i.e. the higher will be the current generated between them in an electrolytic environment. The electric potential or voltage difference between two metals can be measured, but it is not practical to tabulate since it is also a function of the solution involved. It is actually the current generated, not the potential, which causes the corrosion.

It should also be noted that the relative surface areas of the two metals has a bearing on the rate of corrosion as well. The most unfavorable situation is one in which there is a large cathode and a small anode (such as stainless steel fasteners in a copper vessel). In this instance, the corrosion at the anode can be drastically accelerated.

Passivation of metal surfaces with a solution such as dilute nitric acid or nitric acid with an oxidizing salt such as Na_2CrO_7 , is often employed to help minimize corrosion as well, especially following machining, welding or similar operations. In passivation, trace ions of other metals alloyed with the principle metal, or left behind after welding or machining can be largely removed, thereby eliminating another potential source of galvanic action. Passivation also accelerates the formation of an impermeable layer of oxide or other inhibiting compound on the metal surface that greatly reduces its anodic corrosion rate. The best passivation conditions vary from metal to metal. Many metals form this protective layer naturally in air or in aqueous solutions. These are considered **passive** metals, to distinguish them from metals lacking this layer, which are called **active** metals.

Corrosion units – Rates of corrosion of materials are measured in a variety of units, most typically mm/yr (mm/annum) or inches/yr (ipy) which indicate the linear rate of disappearance of surface material. Other units quantify corrosion rates by the mass of material lost per unit surface area. Conversions between some of the more common units are given in the table at the bottom of the page. To use these factors, multiply the starting units by the factor in the table. For example, to convert ipy to mils/year, multiply ipy by 1000.

Galvanic Series of Metals (in Seawater)



Sources: [20, 88, 105]

Corrosion Units Conversion Factors

Corrosion Units	mdd	$\text{g/m}^2/\text{d}$	μ/yr	mm/yr	mils/yr	ipy
mg / square decimeter / day (mdd)	1	0.1	$36.5/\rho$	$0.0365/\rho$	$1.144/\rho$	$0.00144/\rho$
grams / square meter / day ($\text{g/m}^2/\text{d}$)	10	1	$365/\rho$	$0.365/\rho$	$14.4/\rho$	$0.0144/\rho$
microns / year (μ/yr)	$0.0274 \times \rho$	$0.00274 \times \rho$	1	0.001	0.0394	0.0000394
millimeters per year (mm/yr)	$27.4 \times \rho$	$2.74 \times \rho$	1000	1	39.4	0.0394
mils / year (mils/yr)	$0.696 \times \rho$	$0.0696 \times \rho$	25.4	0.0254	1	0.001
inches / year (ipy)	$696 \times \rho$	$69.6 \times \rho$	25,400	25.4	1000	1

ρ = density of material in g/cm^3

Sources: [139, 194]

Properties and Corrosion of Glass

Typical Properties of Borosilicate Glass

Density	Coefficient of Expansion	Softening Temperature	Specific Heat	Thermal Conductivity
2.5 g/cm ³ 156 lb/ft ³	~0.0001/°C ~0.00005/°F	570°C 1058°F	835 J/kg-K 0.2 BTU/lb-°F	1.2 W/m-K 0.69 BTU/lb-°F

Sources [55, 66, 199]

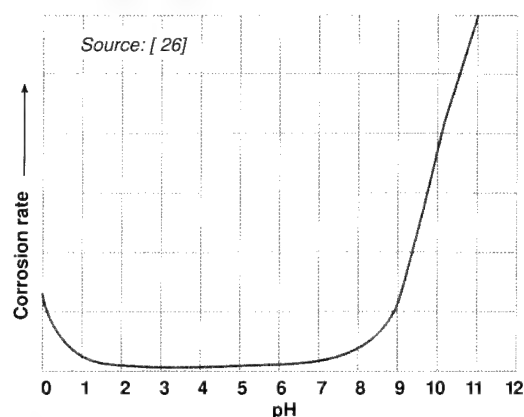
Glass is one of the most chemically inert substances available, and borosilicate glass (typically ~80% SiO₂, ~12% B₂O₃, with Na₂O, Al₂O₃ and other trace compounds) is particularly useful in the chemical processing industry because of its relatively low melting point which allows its use in glass-lined steel chemical reactors. At moderate temperatures, borosilicate glass is inert to almost all substances except hydrofluoric acid (aqueous and gaseous). At high temperatures, phosphoric and other acids and some alkalis can also cause corrosion, but at much less significant rates. A number of specialty glasses are available that exhibit superior corrosion resistance under certain specific conditions and over wider temperature ranges. Options should be discussed in detail with a qualified equipment manufacturer.

Effect of pH – Because corrosion of glass in aqueous solutions is a combination of effects, including the exchange of ions in the glass with ions in the water and the ionization and dissolution of silicic acid, aqueous corrosion rates are highly dependent on pH. The graph at right shows the effect of pH on rate of dissolution of soda-lime silicate glass. The highest rates of dissolution occur at the extremes of pH, particularly on the basic, or high pH, side. Other factors include concentration, glass composition and surface to volume ratio.

Isocorrosion charts – The isocorrosion graphs on the following page show the effect of temperature and concentration on the corrosion of typical reactor glass by some common acids and bases. The data reflect corrosion rates for pure acids and bases, but in practice, most process mixtures will contain other substances that can significantly impact corrosion resistance, both negatively and positively. Use these charts as a very general guide only, since there are many glass formulations, each with its own specific properties and corrosion resistance characteristics. Corrosion tests may need to be performed prior to the introduction of new process conditions in glass reactors. Testing can usually be carried out on process samples by the manufacturer. Note that hydrofluoric acid (HF), not included in these charts, is the single most corrosive acid to glass. HF reacts with SiO₂ to form SiF₄ which is volatile, thus driving the reaction forward. Since corrosion can occur at even very low concentrations and mild temperatures, HF should be completely avoided in glass-lined vessels.

The curve for each acid or base indicates the combination of concentration and temperature that will cause the removal of surface glass at the rate of 0.2 mm/yr. This may be considered an acceptable limit for corrosion rate under most circumstances, but again, this depends on the duty cycle and actual operating conditions. Short term use under these conditions may, at a minimum, cause etching of the glass surface. Conditions above the curves will cause accelerated corrosion; conditions below the curves are milder. Bear in mind that the thickness of glass in a typical reactor is roughly 2 mm. More information on glass-lined reactor vessels can be found in Chapter 2.

Effect of pH on Glass Corrosion Rate

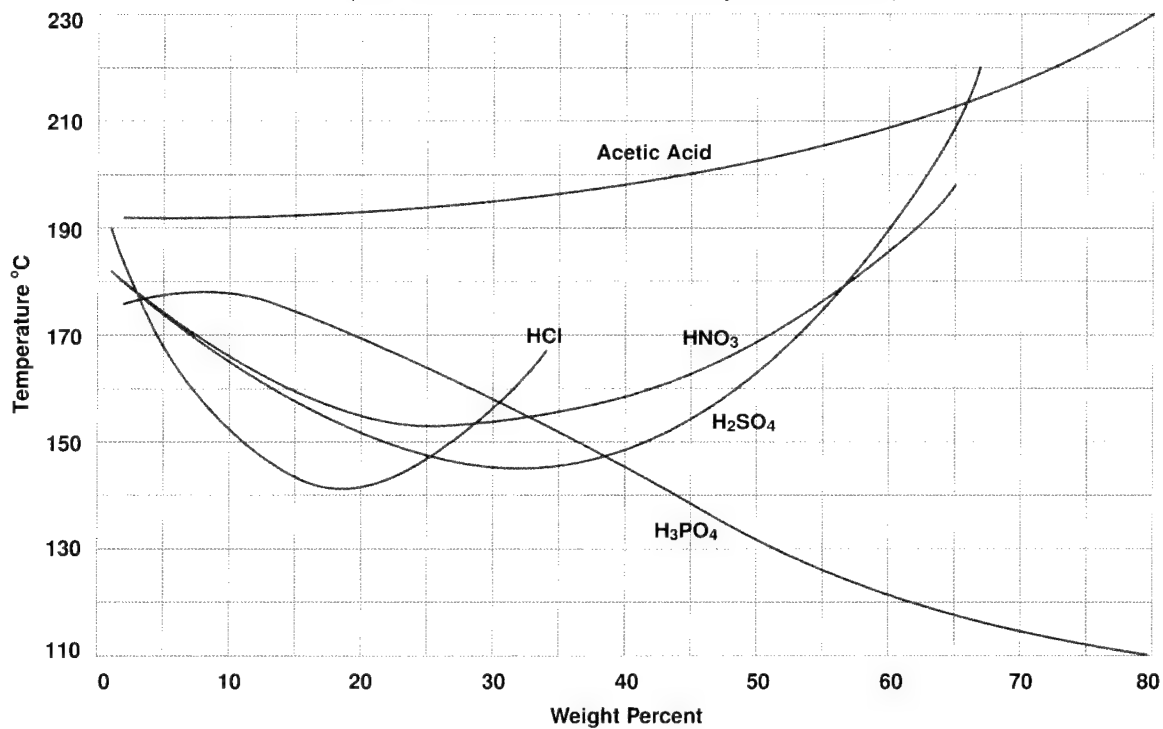


Substances Known to Attack Borosilicate Glass

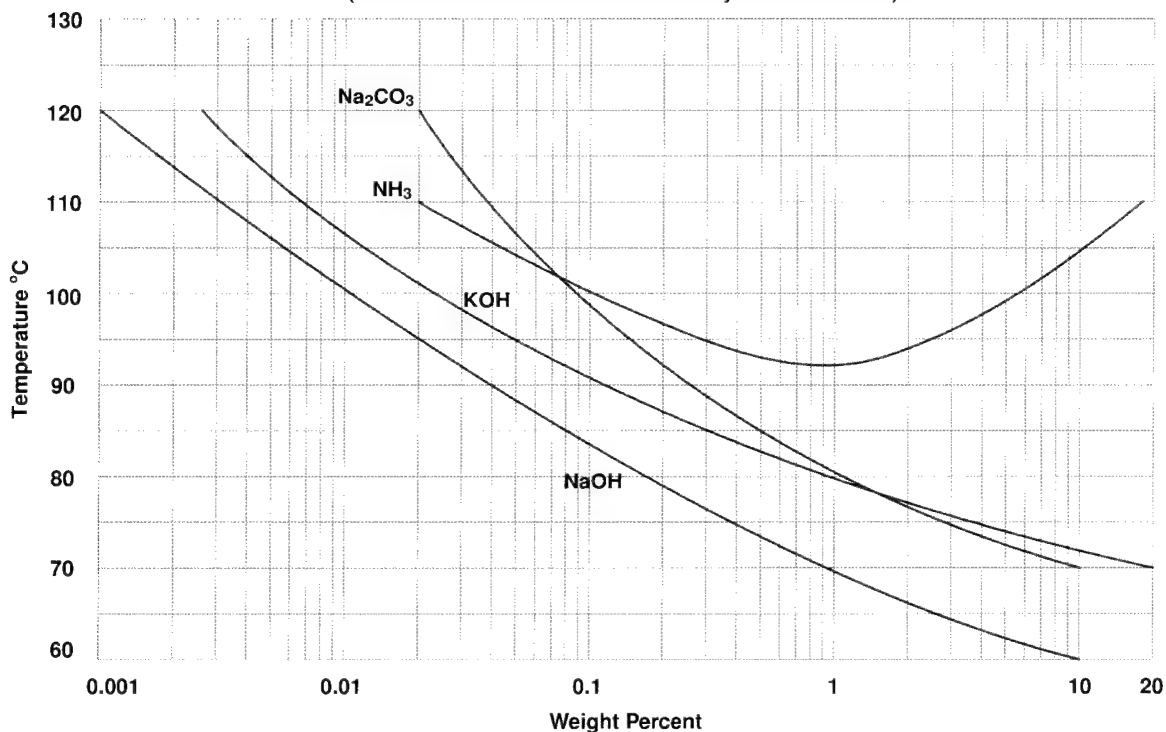
Substance	Concentration	Temp. °C	Substance	Concentration	Temp. °C
Hydrofluoric acid	3%	0	Caustic Potash	molten	360
	1%	20	Hydrochloric Acid	concentrated	120
Sodium Hydroxide	concentrated	boiling	Nitric Acid	concentrated	boiling
Potassium Hydroxide	concentrated	boiling	Phosphoric acid	concentrated	boiling
Caustic Soda	molten	318	Sulfuric Acid	concentrated	230

Sources [55, 66, 199]

Corrosion of Borosilicate Reactor Glass by Common Acids
(Isocorrosion Data Based on 0.2 mm/yr corrosion rate)



Corrosion of Borosilicate Reactor Glass by Common Bases
(Isocorrosion Data Based on 0.2 mm/yr corrosion rate)



Data provided by Pfaunder, Inc. for Type 9100 reactor glass

MATERIAL COMPATIBILITY TABLE	METALS											PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester
Acetaldehyde	C	C	A	A	B	-	A	A	A	-	-	A	D	A	D	D	D	A	A	D	D
Acetamide	D	C	B	A	A	-	B	D	-	-	-	-	D	A	-	-	-	A	A	D	-
Acetates	D	A	B	A	A	A	A	C	A	A	A	A	-	A	-	B	C	A	A	-	-
Acetic Acid	D	C	B	A	B	B	D	C	D	A	-	A	D	D	D	D	C	C	D	D	-
Acetic Acid 10%	D	C	B	A	B	B	D	C	B	A	-	A	C	C	B	A	A	D	C	A	-
Acetic Acid 50%	D	C	B	A	B	B	D	C	B	A	-	A	D	D	D	B	B	B	D	D	A
Acetic Acid 80%	D	C	D	B	B	B	D	C	B	A	-	A	D	D	-	D	C	B	D	B	B
Acetic Anhydride	D	D	A	A	A	B	D	C	B	A	-	A	C	D	-	D	D	C	D	D	A
Acetone	A	A	A	A	A	A	A	A	A	A	A	A	D	A	D	D	D	B	A	D	D
Acetonitrile	B	A	A	A	A	B	-	-	-	-	A	-	D	-	-	D	D	B	-	-	-
Acetyl Chloride	B	B	C	A	D	A	D	-	B	A	-	-	D	D	-	C	C	D	B	D	-
Acetylene gas	A	A	A	A	A	D	B	C	A	-	-	-	-	A	-	A	C	A	A	D	-
Acrylonitrile	A	A	A	C	B	A	A	-	A	B	-	-	D	-	-	D	A	A	A	D	-
Allyl Alcohol	A	A	A	A	B	B	-	-	-	-	-	-	-	-	-	D	-	A	-	D	-
Allyl Chloride	-	-	B	A	D	-	-	-	-	-	-	-	D	-	-	D	D	A	-	-	-
Alum $\text{AlNH}_4(\text{SO}_4)_2$	-	-	-	A	-	-	-	-	-	A	-	-	-	-	-	-	-	-	-	-	A
Aluminum Acetate	C	C	A	A	B	A	-	-	C	A	A	-	-	-	-	A	A	A	-	-	-
Aluminum Chloride	D	B	D	D	D	-	D	D	B	A	A	B	A	C	A	A	A	A	B	A	-
Aluminum Hydroxide	A	D	A	A	B	D	B	C	B	B	-	B	B	A	-	A	A	B	A	B	-
Aluminum Nitrate	-	-	A	A	D	-	-	-	C	-	-	A	-	B	-	B	A	A	A	-	-
Aluminum Potassium Sulfate 10%	D	A	A	A	C	A	A	-	B	C	A	A	-	C	-	A	B	A	A	A	-
Aluminum Potassium Sulfate 100%	D	A	D	B	C	B	D	-	C	C	A	A	-	C	-	A	B	A	D	A	-
Aluminum Sulfate	D	D	C	B	B	A	B	B	C	B	A	A	A	B	A	A	A	A	B	-	-
Amines	D	B	A	A	B	-	B	D	B	B	-	B	-	D	-	C	D	A	A	D	-
Ammonia gas	A	A	B	A	A	D	D	D	C	B	-	C	D	D	-	A	A	A	A	D	-
Ammonia, anh. liquid	A	B	B	A	A	-	D	D	B	B	-	C	D	D	-	B	A	A	-	D	-
Ammonia, aqueous	A	A	A	A	A	-	D	D	B	A	-	C	-	D	D	A	A	A	A	D	-
Ammonium Acetate	A	A	A	A	A	-	D	D	B	-	-	-	-	-	-	B	A	-	A	-	-
Ammonium Bifluoride	D	D	D	B	B	-	-	D	-	B	-	-	A	D	-	A	A	A	-	-	-
Ammonium Carbonate	B	B	A	B	B	D	D	D	B	B	A	A	A	D	-	A	A	A	A	-	D
Ammonium Chloride	D	D	A	D	B	D	D	D	B	B	A	B	A	B	-	A	A	A	A	A	-
Ammonium Fluoride	C	B	D	D	D	D	D	D	-	B	-	A	A	C	-	-	A	B	A	-	-
Ammonium Hydroxide 10%	D	C	A	A	-	-	A	-	D	B	-	-	A	C	B	A	-	-	A	B	D
Ammonium Hydroxide 30%	D	C	A	A	B	D	D	D	D	B	-	A	B	C	B	A	A	A	A	C	D
Ammonium Nitrate	B	D	A	A	B	D	D	D	-	B	-	A	-	A	-	A	A	A	B	-	A
Ammonium Oxalate	D	A	A	A	-	C	-	D	B	A	A	-	-	B	-	A	-	A	-	A	-
Ammonium Phosphate, Dibasic	D	D	B	C	B	D	B	D	C	B	A	A	A	B	-	A	A	A	B	A	-
Ammonium Phosphate, Monobasic	D	D	B	C	B	D	D	D	C	B	A	A	-	B	-	A	A	A	A	-	-
Ammonium Phosphate, Tribasic	D	D	B	B	B	D	C	C	C	B	A	A	-	B	-	A	A	A	A	-	-
Ammonium Persulfate	D	A	A	B	D	D	D	-	C	A	A	A	A	D	-	A	A	A	D	-	D
Ammonium Sulphate	D	C	D	B	A	D	D	D	B	B	A	A	A	B	-	A	A	A	D	A	A
Ammonium Sulfite	D	C	B	B	D	D	C	A	D	-	-	-	-	D	-	A	A	-	A	-	-
Ammonium Thiosulfate	D	A	-	-	-	D	D	D	-	-	-	A	-	B	-	-	-	A	-	-	-
Amyl Acetate	C	C	A	A	A	A	A	A	B	A	A	A	D	B	B	D	D	A	C	D	-
Amyl Alcohol	B	C	A	A	B	A	A	A	B	A	-	B	A	A	D	C	A	B	A	C	A
Amyl Chloride	A	A	C	B	A	A	B	A	B	A	A	C	D	A	-	D	C	A	C	-	-
Aniline	C	C	A	B	C	D	D	C	B	B	-	C	D	A	-	D	B	D	C	D	-
Aniline Hydrochloride	D	-	D	D	D	B	D	D	-	D	A	A	D	-	-	B	D	D	D	D	-

This chart is intended only as a general guide to material selection and use. The information was obtained from sources believed to be dependable, but it should not be relied upon as the sole authority on material selection. Considerable variation in resistance properties was found in the literature, perhaps owing to the existence of thousands of proprietary alloys, copolymers and composites, or differences in testing conditions. For specific applications, more information should be sought from suppliers. Materials should be tested for swelling, corrosion or deterioration under conditions that match expected process conditions as closely as possible.

Percentages (%) refer to aqueous solutions. Unless otherwise stated, listings of organic and inorganic salts indicate aqueous solutions of unspecified concentration. Where data are missing from the chart, it is often possible to extrapolate from similar classes of materials, always bearing in mind the caveats mentioned above. The possibility of synergistic effects must always be considered. Additional information on the corrosion of specific materials (glass, 316SS, Hastel-

PLASTICS										ELASTOMERS										G						
Polyethylene HD	Polyethylene LD	Polypropylene	Polystyrene	Polysulfone	PTFE (Teflon)	Ryton	Buna-N - NBR	Chlorobutyl	ECTFE (Halar)	EPDM	ETFE (Tefzel)	FEP	Hypalon	Natural Rubber	Neoprene	Nitrile-PVC	Noryl	PCTFE (Kel-F)	PEEK	PFA	PVDF (Kynar)	Silicone Rubber	TPE (Hytrel)	Tygon (E-3606)	Viton	Borosil. Glass
C	C	B	-	-	A	A	D	A	-	A	A	A	-	B	D	D	-	A	A	A	A	B	B	D	D	D
B	B	A	D	-	A	A	B	-	-	A	-	-	C	D	C	-	-	A	-	-	B	B	D	D	D	D
B	A	A	B	C	A	A	C	B	A	B	A	A	C	D	C	A	A	A	A	A	B	D	D	B	B	C
A	A	A	B	C	A	A	C	-	A	A	A	A	C	D	C	A	A	A	A	A	A	A	D	D	D	C
-	D	D	B	C	A	A	C	-	-	A	-	-	C	C	C	-	A	A	-	-	C	B	C	D	D	A
D	D	A	D	D	A	A	D	B	B	A	B	A	C	B	B	D	D	A	A	A	D	B	C	D	D	D
-	-	-	-	-	A	A	C	A	B	A	-	A	B	A	C	D	D	A	-	-	B	B	C	-	D	D
-	D	D	A	-	A	A	D	A	-	D	-	-	D	B	C	C	-	A	-	-	A	B	C	D	D	A
A	A	B	-	-	A	-	D	D	-	D	-	-	C	-	D	D	-	-	-	-	B	D	-	D	-	C
-	-	-	D	-	A	-	A	A	-	D	-	-	-	-	D	B	D	-	-	-	A	-	-	-	-	B
A	-	A	-	-	A	A	D	-	-	-	-	-	-	-	A	-	-	-	-	-	-	-	-	-	-	A
-	-	A	-	-	A	A	B	-	-	-	-	-	-	A	B	-	-	-	-	-	-	-	-	-	-	A
-	-	A	-	-	A	A	A	-	-	A	-	-	-	-	A	C	-	-	-	-	-	D	-	-	-	A
A	B	B	A	-	A	-	A	A	-	A	A	A	-	A	A	A	A	A	A	A	A	A	-	-	-	A
-	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	B	A	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	A	A	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	A	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	A	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	A	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	A	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	A	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
A	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
B	A	A	-	-	A	-	A	-	-	A	-	-	-	A	A	-	-	A	-	-	-	B	-	-	-	A
-	D	C	C	D	A	A	D	B	A	D	A	A	D	B	D	D	D	A	A	A	B	D	C	D	D	A
A	B	B	D	A	A	A	B	C	-	D	A	A	A	C	D	D	C	A	-	-	A	D	A	C	B	A
D	D	D	-	-	A	-	D	-	-	A	-	-	D	D	D	D	D	A	-	-	A	D	A	C	B	A
D	C	A	-	-	A	A	D	B	A	B	-	-	D	D	D	D	D	A	-	-	B	C	D	D	D	A
-	D	D	-	-	A	-	C	C	-	B	-	-	-	A	D	D	-	-	-	-	B	D	-	D	B	A

loy and Tantalum) can be found on pages 10-8 and 10-28. Table sources: [2, 17, 62, 63, 75, 84, 88, 139, 174, 179, 180, 187, 226, 227, 231, 236, 249, 258, 268].

The following rating system is used in this table:

A - Excellent. Material is acceptable for continuous use over a wide range of temperatures and operating conditions.

B - Good. Materials should be acceptable for use at moderate temperatures and under mild conditions.

C - Fair. Materials should be acceptable for short-term exposure at moderate temperatures only.

D - Unacceptable. Significant degradation is expected to occur. Material should not be used.

**MATERIAL
COMPATIBILITY
TABLE
(continued)**

	METALS											PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester
Antimony Trichloride	-	-	D	D	D	D	D	A	-	-	A	B	A	-	-	A	A	D	D	A	D
Aqua Regia	D	D	D	D	D	D	D	D	-	C	A	A	D	D	-	D	C	D	D	A	D
Argon	A	A	A	A	A	A	A	A	A	A	-	A	A	A	-	A	A	A	A	A	-
Aromatic Hydrocarbons	A	A	-	B	A	-	A	C	B	-	-	-	-	A	-	D	D	A	-	-	-
Arsenic Acid 20%	D	D	A	A	D	A	D	B	D	B	-	B	A	D	-	D	A	A	A	A	-
ASTM Reference No. 1 oil	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-
Barium Carbonate	A	B	A	A	D	A	B	B	B	B	A	A	A	A	-	A	A	A	A	A	A
Barium Chloride	C	C	D	A	D	B	B	B	B	B	A	A	A	A	-	A	A	A	B	A	A
Barium Cyanide	C	A	A	A	C	D	C	C	D	B	A	-	-	B	-	D	D	A	A	-	-
Barium Hydroxide	D	C	C	A	D	-	D	D	B	B	-	B	A	D	-	A	A	A	A	D	D
Barium Nitrate	A	A	B	B	B	B	D	D	-	-	A	A	-	B	-	B	A	A	A	D	-
Barium Sulfate	B	C	A	A	B	B	B	C	B	A	A	B	A	B	-	A	B	A	A	D	-
Barium Sulfide	D	C	B	B	D	D	D	D	C	-	-	A	A	A	-	D	A	B	A	-	-
Benzaldehyde	A	A	A	A	B	B	A	A	B	A	-	A	B	A	D	D	D	D	C	D	D
Benzene	A	B	A	A	B	B	B	A	A	B	-	A	D	A	D	D	D	C	A	D	D
Benzenesulfonic Acid	-	-	B	B	D	-	-	-	-	B	-	B	-	-	-	B	D	B	D	D	-
Benzoic Acid	D	D	A	A	B	-	B	B	B	B	-	A	-	B	D	A	A	A	D	D	A
Benzyl Alcohol	B	-	A	A	B	B	-	A	-	A	-	A	D	A	D	D	A	C	A	D	A
Benzonitrile	-	-	D	D	-	-	-	-	-	C	-	-	-	-	-	-	-	-	-	-	-
Benzyl Chloride	A	A	B	B	D	C	-	D	-	C	-	-	D	A	-	-	-	-	A	-	-
Benzyl Ether	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-
Bleach solutions	D	C	C	C	A	-	B	-	D	A	-	-	B	D	-	A	A	D	D	-	-
Borax	A	C	A	A	B	B	D	B	A	B	-	B	-	B	-	A	A	A	A	-	-
Boric Acid	D	D	A	A	D	B	C	B	B	A	-	A	-	A	B	A	A	A	A	D	A
Boron Trifluoride	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-	-
Brine	C	D	A	D	-	-	B	-	B	A	-	-	-	-	-	-	-	-	A	-	-
Bromine, anhyd. liquid	D	D	D	D	D	D	B	D	A	A	-	D	D	D	D	D	D	D	D	D	-
Bromine dry gas	D	D	D	D	D	D	B	D	A	A	-	D	D	D	D	D	D	D	D	D	D
Bromobenzene	B	B	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Bromotoluene	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Butadiene	B	B	A	A	A	C	C	C	C	C	-	-	-	A	-	B	A	A	B	D	-
Butane	-	B	A	A	A	C	A	C	A	A	-	A	B	A	D	A	C	A	A	D	-
Butanol, 1-	-	C	A	A	B	B	-	A	A	B	-	B	-	A	D	D	A	B	A	D	D
Butanol, 2-	-	-	A	A	-	-	-	-	-	A	-	-	-	-	-	D	-	-	A	-	D
Butanol, t-	-	-	A	A	-	-	-	-	-	A	-	-	-	-	-	D	-	-	A	-	D
Butyl Acetate	A	A	-	C	A	A	A	A	B	A	A	A	-	A	D	D	C	B	A	D	D
Butyl Ether	-	-	-	A	A	-	-	-	-	-	-	-	D	D	-	A	D	A	A	-	C
Butylamine	-	-	-	A	A	-	-	B	-	B	-	B	-	C	-	D	-	B	A	D	-
Butylcarbitol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	B	-	-	-
Butyl Phthalate	-	-	B	-	B	-	-	-	-	B	-	B	-	-	-	-	D	B	A	D	-
Butylene	A	A	B	A	A	D	-	A	D	A	-	-	-	A	-	B	A	A	B	D	-
Butyraldehyde	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-
Butyric Acid	D	D	B	B	B	C	C	D	B	A	-	A	D	A	-	D	D	A	D	D	A
Butyric Anhydride	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Calcium Bisulfate	D	D	-	A	-	-	C	C	D	-	-	-	-	-	-	A	-	A	A	D	-
Calcium Bisulfide	-	-	B	B	C	-	-	C	-	A	A	A	-	D	-	A	A	A	A	-	-
Calcium Bisulfite	D	D	B	A	D	-	-	-	-	B	-	A	-	D	-	A	A	A	A	D	-
Calcium Carbonate	D	D	A	B	D	-	C	A	B	B	A	B	-	A	-	A	A	A	A	C	-
Calcium Chlorate	C	C	B	A	A	A	-	B	-	A	A	-	B	C	-	A	A	-	A	-	A
Calcium Chloride	C	C	A	C	D	B	B	A	B	A	A	A	B	A	B	A	A	A	A	C	A
Calcium Hydroxide	A	C	A	A	C	-	C	D	A	A	A	A	A	C	C	A	A	A	A	D	A
Calcium Hypochlorite 20%	D	-	D	C	D	D	-	D	-	B	-	A	-	D	-	A	B	A	D	D	A
Calcium Nitrate	B	-	C	B	B	-	-	B	-	B	-	B	A	D	B	A	A	A	A	B	-
Calcium Oxide	A	A	A	A	C	-	-	D	-	A	-	A	D	A	-	B	A	A	B	A	-
Calcium Sulfate	A	C	A	A	C	-	C	A	B	B	A	A	C	D	-	A	A	A	A	A	A
Calgon	D	-	A	A	-	-	-	C	-	-	-	-	-	A	-	-	-	A	A	-	-
Carbitol	D	-	-	-	A	D	D	B	-	A	-	A	D	D	-	A	A	C	A	-	-
Carbon Bisulfide	-	B	A	B	B	-	-	B	B	-	-	-	-	A	-	D	D	A	A	-	-
Carbon Dioxide	D	A	A	A	B	A	B	A	A	A	-	A	B	A	-	A	A	A	-	-	A
Carbon Dioxide (wet)	D	-	A	A	A	-	-	A	-	A	-	A	B	A	-	-	A	A	-	-	-

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

**MATERIAL
COMPATIBILITY
TABLE
(continued)**

	A	C	B	A	A	-	-	D	-	B	-	B	-	A	-	D	D	C	A	A	D	-	D
Carbon Disulfide	A	A	B	A	A	A	A	A	A	B	-	-	-	A	-	A	A	A	A	A	-	-	A
Carbon Monoxide	A	A	B	A	A	A	A	A	A	A	-	-	-	A	-	A	A	A	A	A	-	-	A
Carbon Tetrachloride	D	B	B	B	D	A	A	A	A	A	A	A	D	B	-	D	D	A	A	A	D	-	B
Carbonated Water	B	B	A	A	A	B	B	A	B	-	-	-	-	A	-	A	A	A	A	A	-	-	-
Carbonic Acid, liquid	D	-	A	B	B	-	D	B	-	A	-	B	-	B	D	A	A	A	A	A	D	-	-
Cellosolve	B	B	B	B	B	B	-	-	-	-	-	-	-	A	-	-	-	C	-	-	-	-	
Cetyl Alcohol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	A	-	D	-	
Chloric Acid	D	-	D	C	D	D	D	D	-	A	-	-	-	D	-	D	A	-	-	-	-	-	
Chlorine gas (dry)	D	D	C	A	C	B	D	B	A	A	-	D	-	D	D	A	D	D	-	-	D	-	
Chlorine gas (wet)	D	D	D	D	D	D	D	B	A	C	-	A	-	D	D	A	A	A	A	D	D	D	
Chlorine, anhydr. liquid	D	D	B	B	D	-	D	D	-	D	-	D	-	A	-	D	D	C	D	D	C	D	
Chloroacetic Acid 20%	D	-	B	A	D	D	D	C	-	A	-	A	-	D	-	D	D	C	D	D	D	-	
Chlorobenzene	B	C	A	A	A	B	B	C	B	A	-	B	D	D	-	D	D	C	C	A	D	-	
Chlorobromomethane	B	-	-	-	-	B	-	-	-	-	-	-	-	-	-	D	-	-	C	-	-	-	
Chloroform	B	B	A	A	B	A	B	B	A	A	-	A	D	A	-	D	D	C	C	D	-	-	
Chlorosulfonic Acid	D	B	D	D	C	D	B	D	B	A	-	A	-	D	-	D	D	C	D	C	-	-	
Chlorotoluene	A	A	B	B	D	C	-	D	-	C	-	-	D	A	-	-	-	-	A	-	-	-	
Chromic Acid 20%	D	D	B	B	D	D	D	D	C	D	-	A	B	D	B	A	A	B	D	C	-	D	
Chromic Acid 50%	D	D	B	B	D	D	D	D	D	B	-	A	D	D	D	A	D	C	D	D	D	D	
Chromic Acid 80%	D	D	B	B	D	D	D	D	D	C	-	-	D	D	D	-	D	D	D	D	D	D	
Citric Acid	D	D	A	A	C	D	D	D	B	A	-	A	D	B	B	A	B	A	C	B	-	A	
Citric Oils	D	-	A	A	C	-	-	A	-	-	-	-	-	B	-	-	-	A	-	-	-	-	
Copper Chloride	-	-	D	B	-	-	-	D	-	-	A	D	A	A	-	A	A	A	D	-	-	A	
Copper Cyanide	A	-	A	A	D	-	D	D	C	A	A	B	-	A	-	A	A	B	A	D	B	-	
Copper Nitrate	D	D	A	A	D	D	D	D	D	B	A	B	-	A	-	A	A	A	D	D	-	-	
Copper Sulfate	D	D	B	B	D	-	D	D	C	A	A	A	-	D	-	A	A	A	D	A	-	A	
Cottonseed Oil	A	C	A	A	A	A	A	-	B	A	-	A	A	A	-	C	A	A	A	D	-	-	
Cresol (o-, m-, p-)	C	-	C	C	A	A	-	A	-	B	-	B	D	D	-	C	D	A	-	D	-	-	
Cupric Chloride	-	-	-	D	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	D	-	-	
Cupric Nitrate	D	D	A	A	D	D	D	-	D	-	A	-	-	-	-	-	-	-	-	-	-	-	
Cupric Sulfate	D	D	A	A	D	D	D	-	-	-	-	-	-	-	-	-	A	-	-	D	-	-	
Cresylic Acid	D	C	A	A	B	B	C	D	B	B	-	A	-	D	-	B	D	D	D	D	-	-	
Cupric Acid	-	-	D	B	D	-	-	-	-	A	-	A	-	-	-	A	-	A	D	A	-	-	
Cyanic Acid	D	-	A	A	-	-	-	-	-	-	-	-	-	D	-	-	-	A	-	-	-		
Cyclohexane	B	A	A	A	A	B	A	B	B	B	-	A	B	A	D	D	D	A	-	D	-	-	
Cyclohexanol	B	B	B	B	D	B	-	-	-	-	-	-	B	-	-	-	-	B	-	-	-	-	
Cyclohexanone	B	-	A	A	A	B	B	B	B	A	-	-	D	A	-	D	D	C	A	D	-	-	
Cyclopentane	-	-	A	A	A	B	A	B	B	B	-	A	-	-	-	-	-	-	-	-	-	-	
Decane	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	B	-	-	-	-	
Detergents	B	A	A	A	B	-	B	B	B	B	-	A	B	A	-	A	A	A	A	A	-	-	
Dextrose	-	-	A	A	B	B	B	B	-	A	-	-	A	-	-	A	A	A	A	-	-	-	
Diacetone Alcohol	A	A	B	B	A	A	A	A	B	A	-	A	-	A	-	D	D	A	A	D	-	-	
Diborane	-	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	-	-	-	-	
Dibutyl Phthalate	A	A	A	A	A	A	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-	
Dibutylamine	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	C	-	-	-	-	
Dichlorobenzene, 1,2-	A	A	A	A	C	-	-	B	-	A	-	-	D	-	-	D	D	A	-	D	D	-	
Dichloroethane, 1,2-	-	-	A	A	B	A	B	D	B	A	-	B	D	A	-	D	D	D	A	-	-	-	
Dichloroethylene	-	-	A	A	C	-	-	-	-	-	-	-	-	D	D	-	-	-	-	D	-	-	
Dichloromethane	B	-	A	A	C	B	A	B	-	B	-	B	D	B	D	D	D	A	C	D	-	-	
Diesel Fuel	A	A	A	A	A	A	A	A	A	B	-	B	-	A	-	A	A	A	A	A	-	-	
Dieth. Glycol Ethyl Ether Acetate	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Diethanolamine	A	B	A	A	A	C	-	-	A	A	A	A	B	-	-	-	-	B	A	-	-	-	
Diethyl Ketone	-	-	A	A	-	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-	-	
Diethyl Ether	-	-	B	B	B	A	B	A	-	B	-	A	D	-	-	D	D	D	A	D	-	-	
Diethylamine	B	A	A	A	B	A	A	A	A	A	-	A	D	B	-	C	D	A	A	A	D	-	
Diethylene Glycol	A	-	A	A	B	-	-	-	B	B	-	A	B	A	-	A	A	C	A	B	-	-	
Dimethylacetamide, N,N-	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Dimethyl Aniline	-	-	B	B	A	-	-	-	-	B	-	A	D	D	-	D	D	A	A	D	-	-	
Dimethylformamide	C	-	A	B	B	A	B	B	B	A	-	A	D	D	-	D	D	D	A	D	-	-	
Dioxane	B	-	-	B	-	-	B	-	B	-	-	-	-	-	-	-	-	-	-	-	-	-	

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

**MATERIAL
COMPATIBILITY
TABLE
(continued)**

MATERIAL COMPATIBILITY TABLE (continued)	METALS												PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester	
Diphenyl	B	B	B	B	B	B	B	B	-	B	-	B	-	-	-	-	-	-	-	-	-	
Diphenyl Oxide	A	A	A	A	B	A	-	-	-	B	-	A	-	D	-	D	-	A	-	-	-	
Diethylene Glycol Diethyl Ether	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	C	-	-	-	
Diethylene Glycol Ethyl Ether	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	C	-	-	-	
Diethylene Glycol Hexyl Ether	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Diethylene Glycol Methyl Ether	-	-	A	A	-	-	-	-	-	-	-	-	-	-	D	-	-	C	-	-	-	
Diisobutyl Ketone	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Dimethyl phthalate	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	D	-	
Dimethyl Sulfoxide	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	
Dioxane 1,4-	A	A	A	A	B	A	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	
Dipropylene Glycol	-	-	-	A	-	-	-	-	-	-	-	-	-	-	-	-	-	B	-	-	-	
Dipropylene Glycol Methyl Ether	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	D	-	-	D	
Dowtherm HTF's	A	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	C	-	-	
Dynalene HTF's	-	A	A	A	A	A	A	A	A	A	A	A	-	A	-	B	-	A	B	-	-	
Ethane	B	B	A	A	A	A	A	-	A	-	-	-	-	A	-	A	-	A	D	-	-	
Ethanol	B	A	A	A	B	A	A	A	B	A	-	A	B	A	D	D	B	A	A	D	D	
Ethanol (denatured)	B	A	A	A	B	A	A	A	B	A	-	A	B	A	-	A	B	A	A	B	-	
Ethanolamine	B	A	A	A	B	D	-	B	-	B	-	B	-	B	-	D	-	A	A	-	-	
Ethoxyethanol 2- cellosolve	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	
Ethoxyethyl acetate 2-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	
Ethyl Acetate	A	B	A	A	A	A	B	A	B	A	A	A	D	A	D	D	D	A	A	D	-	
Ethyl Benzoate	A	A	A	A	A	A	-	-	-	-	-	-	D	-	-	D	D	-	-	D	-	
Ethyl Bromide	A	A	A	A	A	A	-	-	B	-	-	-	-	-	-	-	-	-	-	-	-	
Ethyl Cellosolve	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Ethyl Chloride	C	B	A	A	B	B	A	A	B	B	A	A	D	A	D	D	D	D	A	D	-	
Ethyl Ether	C	B	A	A	B	A	B	A	A	B	-	A	D	A	D	D	D	A	C	D	D	
Ethyl Sulfate	-	-	D	D	-	B	-	D	-	-	-	-	-	-	-	-	-	A	-	-	-	
Ethyl formate	A	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Ethylamine	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Ethylbenzene	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Ethylene	A	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	-	-	-	
Ethylene Bromide	-	-	A	A	B	-	-	B	-	B	-	B	D	-	-	D	D	-	-	D	-	
Ethylene Chloride	D	C	A	A	B	B	-	A	B	-	A	B	D	A	D	D	D	D	A	D	D	
Ethylene Chlorohydrin	-	-	B	B	B	B	B	B	-	B	-	B	D	D	-	D	D	D	D	D	-	
Ethylene Diamine	-	-	B	B	B	D	D	B	-	C	-	A	D	D	-	D	D	A	D	A	-	
Ethylene Dichloride	A	C	B	B	A	-	B	C	B	B	-	B	D	B	-	D	D	D	-	D	-	
Ethylene Glycol	A	C	A	A	A	A	B	A	B	B	-	A	A	B	D	A	A	A	-	D	A	
Ethylene Glycol Butyl Ether	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	C	-	D	-	
Ethylene Glycol Methyl Ether	B	B	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	C	-	-	-	
Ethylene Oxide	D	B	A	A	D	D	D	C	A	A	-	-	D	D	D	A	C	D	A	B	-	
Ethylene trichloride	A	A	A	A	D	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Fatty Acids	C	D	A	A	A	D	C	A	B	A	-	B	A	A	-	D	A	A	A	B	-	
Ferric Chloride	D	-	D	D	D	D	D	D	-	B	A	A	A	D	B	A	A	A	D	B	A	
Ferric Nitrate	D	D	A	A	D	D	D	C	D	B	A	A	A	D	-	A	A	A	D	A	A	
Ferric Sulfate	D	D	A	C	D	D	D	C	D	A	A	A	A	D	B	A	A	A	A	A	-	
Ferrous Chloride	D	D	D	D	D	B	D	C	D	B	A	A	A	D	-	A	A	A	D	D	A	
Ferrous Sulfate	D	D	A	C	B	B	B	B	B	B	A	A	A	D	-	A	A	A	D	A	A	
Flourine gas	D	-	D	D	A	C	A	C	A	B	-	D	A	D	-	D	D	A	D	C	-	
Flourine liquid	-	-	D	D	-	-	-	-	-	-	-	-	-	A	-	-	-	-	D	-	-	
Fluoboric Acid 48%	D	-	B	B	D	-	-	B	-	A	-	D	A	A	-	A	A	D	C	-	B	
Fluosilicic Acid	D	D	C	B	D	D	B	B	A	B	-	D	A	A	-	A	A	C	D	A	D	
Fluosilicic Acid 25%	-	D	-	B	-	-	-	-	A	-	-	-	-	-	-	A	-	-	D	-	D	
Formaldehyde 100%	C	C	C	A	A	A	B	B	B	A	-	A	B	A	-	A	A	B	D	A	-	
Formaldehyde 37%	B	B	-	A	B	B	A	A	B	B	-	B	A	A	C	A	A	A	A	-	-	
Formamide	B	B	B	B	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Formic Acid	D	D	A	D	A	C	D	C	B	A	-	C	D	A	C	B	A	C	D	D	D	
Formic Acid 25%	D	D	A	B	-	-	B	-	B	-	-	-	-	D	C	A	-	-	D	D	-	
Freon 11	A	B	A	A	D	A	B	-	B	A	-	B	D	D	D	A	A	A	D	D	-	
Freon 12	A	A	A	B	D	A	B	B	B	A	-	B	A	B	D	A	A	A	A	D	-	
Freon 22	A	A	A	A	D	B	A	-	-	A	-	B	-	A	D	A	B	A	A	A	D	

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

THE PILOT PLANT REAL BOOK

**MATERIAL
COMPATIBILITY
TABLE
(continued)**

MATERIAL COMPATIBILITY TABLE (continued)	METALS												PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester	
Freon 113	A	A	A	A	D	A	B	-	B	A	-	C	-	A	-	C	B	A	A	B	-	
Freon TF	A	A	A	A	D	A	B	-	B	A	-	B	-	A	-	B	B	A	C	-	-	
Fructose	C	D	A	A	A	-	B	-	B	A	-	-	-	-	-	A	A	-	A	-	-	
Fuel Oils	A	B	A	A	C	A	B	A	B	A	-	A	D	A	-	A	-	A	A	B	-	
Furan	-	A	A	A	A	-	-	-	-	B	-	-	-	D	-	-	-	-	-	-	-	
Furfural	B	A	A	A	A	A	-	B	B	B	-	-	D	A	-	D	D	A	-	D	D	
Furfuryl Alcohol	A	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Gallic Acid	D	D	A	B	D	D	C	B	B	B	-	B	-	-	-	A	C	-	A	-	-	
Gas, natural	B	B	A	A	-	-	B	-	A	-	-	-	-	-	-	-	-	-	A	-	-	
Gasoline	-	A	A	A	A	B	A	A	B	A	-	A	D	A	C	C	-	A	A	D	B	
Gasoline (unleaded)	A	A	A	A	A	B	A	A	A	A	-	A	D	A	-	C	C	A	A	A	-	
Glucose	A	B	A	A	A	A	A	-	A	A	-	A	B	A	-	A	A	B	A	A	-	
Glycerol	A	B	A	A	A	A	B	A	A	A	-	A	C	A	D	A	A	A	A	B	A	
Glycolic Acid (aq)	-	-	A	A	-	-	-	-	-	A	-	A	B	A	-	B	A	A	-	C	-	
Gold Monocyanide	D	-	A	A	-	-	-	-	-	-	-	-	-	A	-	-	-	A	-	-	-	
Helium	B	A	A	A	A	A	A	-	A	A	-	-	-	-	-	-	-	-	-	-	-	
Heptane	A	B	A	A	A	A	A	A	B	A	-	A	D	A	A	D	A	A	A	B	A	
Hexane	A	B	A	A	A	A	A	A	B	A	-	A	D	A	C	D	B	A	A	C	-	
Hexanol 1-	B	A	-	A	B	A	A	A	A	A	-	A	B	A	-	-	-	A	A	-	-	
Hexylene Glycol	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	-	
Hydraulic Fluids	A	A	A	A	A	A	A	A	A	A	-	-	-	B	-	A	-	A	A	C	-	
Hydraulic Oil (synthetic)	A	A	A	A	A	A	A	A	-	A	-	-	-	-	-	A	-	A	A	-	-	
Hydrazine	D	D	A	A	-	A	D	-	D	-	-	-	-	B	-	D	D	A	-	D	-	
Hydrobromic Acid	D	D	D	D	D	D	D	-	-	-	-	A	-	C	-	A	A	B	D	-	A	
Hydrobromic Acid 20%	D	D	D	D	-	-	-	-	-	B	-	-	-	-	-	A	-	-	D	-	A	
Hydrobromic Acid 50%	D	D	D	D	-	-	-	-	-	B	-	-	-	-	-	A	-	-	D	-	A	
Hydrochloric Acid 10%	D	D	D	D	D	D	-	D	-	A	-	D	A	C	B	A	A	A	D	C	A	
Hydrochloric Acid 37%	D	D	D	D	D	D	-	D	-	B	-	D	A	C	D	A	A	A	D	D	A	
Hydrocyanic Acid	D	C	D	D	A	D	D	A	C	A	-	B	B	B	C	A	A	A	A	A	A	
Hydrofluoric Acid 10%	D	D	C	D	D	B	-	B	-	B	-	D	C	D	C	A	C	A	D	C	-	
Hydrofluoric Acid 48%	D	D	C	D	D	B	-	B	-	B	-	D	C	D	C	A	C	C	D	D	D	
Hydrofluoric Acid 75%	D	D	D	D	D	B	-	B	-	B	-	D	C	D	-	C	C	B	D	D	-	
Hydrofluoric Acid 100%	D	D	B	B	D	B	-	B	-	B	-	D	D	D	-	C	C	-	-	D	-	
Hydrofluoroether HTF's	-	A	A	A	-	A	A	-	A	A	-	-	-	-	A	A	-	-	-	A	-	
Hydrogen Chloride gas	D	D	C	A	D	D	D	A	A	A	-	C	-	-	-	A	A	A	D	-	-	
Hydrofluosilicic Acid 100%	D	D	D	D	D	-	A	B	B	B	-	D	-	A	-	B	-	C	D	-	-	
Hydrofluosilicic Acid 20%	D	D	C	B	D	B	A	B	B	B	-	D	-	B	-	D	A	C	D	-	-	
Hydrogen gas	B	B	A	A	A	A	A	A	A	A	-	A	-	-	-	A	A	-	A	A	-	
Hydrogen Peroxide 10%	C	D	C	B	A	D	C	B	D	A	A	A	A	D	D	A	A	C	D	C	D	
Hydrogen Peroxide 30%	C	D	B	B	A	D	C	B	D	A	A	B	-	D	C	A	A	B	D	B	D	
Hydrogen Peroxide 50%	D	D	B	A	A	D	D	B	D	A	A	A	-	D	-	A	A	-	D	A	-	
Hydrogen Peroxide 90%	D	D	B	A	A	D	D	B	D	A	A	B	A	D	B	C	A	A	D	B	-	
Hydrogen Sulfide	D	B	C	A	B	D	D	B	B	A	A	A	-	B	C	A	A	A	D	B	-	
Hydrogen Sulfide (wet)	D	C	C	A	B	-	D	A	C	A	-	B	B	C	-	A	A	A	D	A	-	
Hydroquinone	-	-	B	B	B	B	-	-	-	B	-	B	D	A	-	B	A	-	D	-	-	
Hydroxyacetic Acid 70%	B	-	-	-	-	-	-	-	-	-	-	-	-	-	-	A	-	A	-	-	D	
Hypochlorous Acid 25%	-	-	-	-	-	-	-	-	-	A	-	-	-	-	-	A	-	-	-	-	-	
Iodine	D	D	D	D	A	D	-	A	-	A	-	A	D	D	D	A	D	C	D	B	-	
Iodoform	C	B	A	A	-	B	C	-	C	D	-	B	-	-	-	A	-	-	A	-	-	
Isoamyl acetate	-	-	A	A	-	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-	
Isooctane	B	A	A	A	A	-	A	A	A	-	-	-	-	-	-	A	-	A	A	B	-	
Isoamyl Alcohol	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	
Isobutane	-	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	-	-	-	
Isobutanol	B	A	A	A	B	A	A	A	-	A	-	A	B	A	-	D	-	A	A	-	-	
Isobutyl Acetate	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	B	-	D	-	
Isobutyl Chloride	B	B	B	B	B	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Isooctane	B	A	-	-	-	-	A	-	A	-	-	-	-	-	-	D	-	-	-	-	-	
Isopropanol	A	A	A	A	-	-	-	A	B	A	-	A	-	A	D	D	C	A	A	C	-	
Isopropyl Acetate	-	-	-	B	D	-	-	A	-	B	-	-	D	D	-	D	-	A	B	D	-	
Isopropyl Ether	B	A	-	A	A	B	A	A	B	A	-	-	-	D	-	D	-	D	A	D	-	

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

MATERIAL COMPATIBILITY TABLE (continued)	METALS											PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester
Isopropylamine	A	A	A	A	-	-	-	-	-	-	-	-	D	-	-	-	-	A	D	-	-
Isotane	-	-	-	-	D	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	-
Kerosine	A	B	A	A	A	B	A	A	A	B	-	A	D	A	C	A	-	A	A	D	-
Ketones	A	A	A	A	B	A	A	A	A	A	-	A	A	D	D	D	-	C	A	D	-
Krypton	-	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	-	-	-
Lactic Acid 10%	D	C	A	A	B	B	D	-	C	B	A	-	-	B	B	A	-	-	C	A	A
Lactic Acid 85%	D	D	A	B	B	B	D	B	D	B	A	A	D	B	B	D	A	B	C	B	-
Laquer Thinner	C	C	A	A	A	A	A	A	A	A	-	C	A	D	-	D	-	A	A	B	-
Lacquers	C	C	A	A	A	A	A	A	A	A	-	C	A	D	-	D	-	A	A	D	-
Lead Acetate	A	D	A	A	D	-	C	B	B	B	A	A	B	B	-	A	A	A	-	-	A
Lead Nitrate	-	-	B	A	D	-	-	B	-	B	-	-	B	-	-	A	A	-	-	-	-
Lead Sulfamate	-	-	C	C	C	-	-	-	-	-	-	-	-	A	-	B	-	A	B	A	-
Lemon Oil	D	D	A	A	C	-	-	A	-	-	-	-	-	B	-	D	-	A	D	-	-
Ligroin	-	-	-	A	D	-	-	-	-	-	-	-	-	B	-	-	-	A	-	-	-
Limonene-D	-	-	A	A	A	-	-	A	-	A	A	-	C	-	-	D	A	A	A	-	-
Linoleic Acid	B	B	B	A	A	D	B	-	B	-	-	-	A	B	-	C	A	-	-	-	-
Linseed Oil	A	A	A	A	-	-	B	-	B	A	-	-	A	-	-	C	C	B	A	-	A
Lithium bromide	A	A	-	-	-	-	-	-	-	-	-	-	-	A	-	-	-	-	-	-	-
Lithium Chloride	A	-	A	A	D	-	B	A	B	-	-	-	-	A	-	D	A	-	B	-	-
Lithium Hydroxide	A	A	B	B	D	-	-	B	-	B	-	-	-	-	-	-	-	-	D	-	-
Magnesium Bisulfate	-	B	A	A	D	-	B	A	B	-	-	-	-	-	-	A	-	-	A	A	-
Magnesium Carbonate	B	B	A	A	A	A	B	A	B	B	-	A	B	A	-	A	A	A	-	A	A
Magnesium Chloride	D	C	B	A	D	A	C	B	B	A	A	A	B	B	-	A	A	A	A	B	A
Magnesium Hydroxide	A	B	A	A	C	B	C	B	B	A	A	A	B	A	-	A	A	A	A	-	-
Magnesium Nitrate	D	-	A	A	B	B	-	A	B	A	A	A	B	A	-	A	A	A	A	A	-
Magnesium Oxide	A	-	A	A	B	-	-	-	-	-	-	-	-	A	-	-	-	A	-	-	-
Magnesium Sulfate	A	B	B	A	B	A	A	A	B	B	A	A	B	B	-	A	A	A	A	B	A
Maleic Acid	B	B	A	A	B	-	B	B	B	B	-	A	-	A	-	D	A	A	-	-	-
Maleic Anhydride	B	-	A	A	A	-	B	-	B	-	-	-	-	D	-	-	-	A	-	-	-
Malic Acid	D	D	A	A	B	D	B	B	B	B	-	A	-	A	-	A	-	-	A	-	-
Manganese Sulfate	A	-	B	B	B	B	D	A	-	A	-	A	B	A	-	C	A	-	A	A	-
Melamine resin	D	-	-	D	-	-	-	-	-	-	-	-	-	A	-	D	A	A	A	-	-
Mercuric Chloride	D	D	D	D	D	D	D	D	D	C	A	A	B	B	-	A	A	A	A	B	A
Mercuric Cyanide	C	D	C	C	D	D	D	D	C	A	A	A	B	-	-	A	A	A	A	-	-
Mercuric Nitrate	-	-	A	A	D	-	D	-	D	A	-	A	C	-	-	A	A	-	-	-	-
Mercury	A	A	A	A	D	D	D	A	B	A	-	A	B	A	-	A	A	A	A	D	-
Methacrylic acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Methane gas	B	B	A	A	A	A	A	A	B	A	-	-	-	A	B	A	-	-	A	D	-
Methanol	A	A	A	A	A	B	A	A	A	A	-	B	D	A	C	D	A	B	A	D	D
Methanolamine	-	B	A	A	-	B	-	-	A	A	A	A	-	-	-	-	-	A	A	-	-
Methyl Acetate	A	B	A	A	A	B	A	A	B	A	-	-	D	B	-	D	-	D	A	D	-
Methyl Acetone	A	A	A	A	A	-	A	A	A	-	-	-	-	D	-	D	-	C	A	-	-
Methyl Acrylate	A	-	A	-	-	-	-	-	-	-	-	-	-	B	-	-	-	A	-	-	-
Methyl Amine	A	B	A	A	A	-	D	A	C	-	-	-	D	D	-	D	-	A	-	-	-
Methyl Bromide	C	-	A	A	D	B	C	-	B	-	-	-	D	D	-	D	D	B	D	-	-
Methyl Butyl Ketone	-	-	A	A	-	-	-	-	-	-	-	-	-	D	-	-	-	C	D	D	-
Methyl Cellosolve	C	B	A	A	B	B	A	A	B	-	-	-	-	D	-	D	D	C	C	D	-
Methyl Chloride	D	A	A	A	D	A	B	B	A	B	-	A	D	B	D	D	D	A	A	D	-
Methyl Dichloride	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	A	-	A	C	-	-
Methyl Ethyl Ketone	A	A	A	A	B	A	A	A	A	A	-	A	D	C	D	D	D	C	A	D	D
Methyl Formate	C	C	-	B	-	-	A	-	B	-	-	-	-	-	-	-	-	-	-	-	-
Methyl Iodide	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Methyl Isobutyl Ketone	C	-	B	A	B	B	-	A	-	A	-	A	D	-	-	D	D	C	A	D	-
Methyl Isopropyl Ketone	C	-	A	A	A	A	-	A	-	-	-	-	-	-	-	D	-	A	A	D	-
Methyl Methacrylate	C	-	B	B	-	-	-	-	-	-	-	-	-	D	-	C	-	A	-	-	-
Methylene Chloride	B	B	B	B	C	B	A	B	B	B	-	B	D	B	-	D	D	A	D	D	-
Methyl propyl ketone	-	-	-	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Methyl t-Butyl Ether	-	-	-	A	-	-	-	-	-	-	-	-	-	-	-	D	-	-	A	D	-
Methylene bromide	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Mineral Spirits	B	B	A	A	A	-	B	A	B	B	-	B	D	A	-	C	A	A	A	C	-

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

PLASTICS														ELASTOMERS												G
Polyethylene HD	Polyethylene LD	Polypropylene	Polystyrene	Polysulfone	PTFE (Teflon)	Ryton	Buna-N - NBR	Chlorobutyl	ECTFE (Halar)	EPDM	ETFE (Tezel)	FEP	Hypalon	Natural Rubber	Neoprene	Nitrile-PVC	Noryl	PCTFE (Kel-F)	PEEK	PFA	PVDF (Kynar)	Silicone Rubber	TPE (Hytel)	Tygon (E-3606)	Viton	Borosil. Glass
-	-	-	-	-	A	-	D	-	-	-	-	-	-	-	D	-	-	-	-	-	A	-	-	-	D	-
D	C	C	D	D	A	A	A	-	-	D	-	A	D	D	D	D	D	A	A	A	A	D	D	D	A	-
-	-	A	-	B	A	A	D	A	-	A	A	A	A	A	D	A	A	B	A	A	B	A	B	B	A	A
A	A	A	-	-	A	-	D	D	-	D	-	-	D	D	A	D	A	-	A	A	D	D	D	D	D	-
D	A	A	-	-	A	-	D	D	-	D	-	-	D	D	D	D	B	A	-	-	-	D	D	D	D	-
A	A	A	A	-	A	A	B	A	-	A	-	-	-	A	D	-	A	-	-	-	A	A	B	A	A	A
-	B	-	-	-	A	-	B	-	-	C	-	-	C	B	D	-	-	-	-	-	A	C	-	-	-	-
D	A	D	-	-	A	A	C	D	-	D	-	-	D	D	D	D	-	-	A	-	A	D	-	-	-	-
-	-	A	-	-	A	B	A	B	-	C	-	-	C	D	D	D	-	-	-	-	A	B	-	-	-	-
D	-	-	-	-	A	-	A	A	-	A	-	-	-	D	D	C	A	-	-	-	A	A	B	B	A	-
-	-	-	-	-	A	-	C	-	-	-	-	-	-	-	A	-	-	-	-	-	-	-	-	A	-	B
A	B	A	A	-	A	A	A	-	-	-	-	-	A	-	B	-	-	-	-	-	-	-	-	-	-	D
A	A	A	-	-	A	A	A	-	-	A	-	A	A	A	A	A	-	-	A	-	A	C	A	-	A	A
A	A	A	-	-	A	A	B	A	-	A	A	A	A	A	A	A	A	-	A	A	A	A	A	A	A	-
-	A	A	-	-	A	A	A	A	-	A	A	A	A	-	A	A	A	-	A	A	A	A	B	B	A	-
A	A	A	A	-	A	B	A	D	-	A	A	A	A	D	D	D	A	-	-	A	A	-	-	-	-	A
A	D	D	-	-	A	-	D	B	-	D	-	-	D	D	D	A	-	-	-	-	A	A	-	-	-	A
-	-	A	-	-	A	A	C	A	-	A	-	-	A	-	A	-	-	A	-	-	-	C	-	-	-	-
A	B	A	A	-	A	A	A	-	-	A	-	A	A	-	-	-	A	-	A	-	A	-	-	-	-	-
-	A	A	-	-	A	A	B	-	-	A	A	A	A	B	B	-	A	-	-	A	A	A	A	A	A	-
-	-	B	-	-	A	A	A	A	-	A	-	A	A	-	A	-	A	-	-	A	A	-	-	-	-	-
A	A	B	B	B	A	A	A	A	B	A	A	A	A	A	A	-	A	A	A	A	A	A	A	A	A	-
-	C	B	D	-	A	-	D	A	-	A	A	A	-	D	D	-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	A	-	D	-	-	B	-	-	D	A	D	A	-	-	-	-	A	-	-	-	-	-
-	-	D	-	-	A	-	D	-	-	A	-	-	-	-	-	-	-	A	-	-	-	-	-	-	-	-
-	-	A	C	-	A	-	C	D	-	B	-	-	-	D	D	-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	A	-	D	-	-	B	-	-	-	D	D	-	-	-	-	-	-	-	-	-	-	-
-	-	C	D	-	A	-	D	-	-	D	A	-	-	D	D	-	-	-	-	-	-	-	-	-	-	-
-	-	D	-	-	A	B	D	-	-	D	-	A	-	-	D	-	-	A	A	A	-	-	-	-	-	-
-	-	B	-	-	A	A	D	A	A	C	A	A	-	-	D	-	-	-	-	-	D	-	-	-	-	-
-	-	-	-	-	A	-	D	-	B	A	-	-	B	-	B	-	-	-	-	-	-	-	-	-	-	-
D	C	C	D	-	A	A	D	D	A	C	-	-	D	D	D	D	D	A	-	-	-	C	-	-	-	-
-	-	D	-	-	A	-	D	-	-	D	-	-	-	D	D	-	-	-	-	-	B	-	-	-	-	-
-	-	D	-	-	A	-	D	-	A	C	-	-	-	D	D	-	-	-	-	-	-	-	-	-	-	-
-	-	-	-	-	A	-	D	-	-	B	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
C	D	C	D	D	A	-	D	-	A	D	A	A	-	-	-	-	-	A	-	A	A	-	-	-	-	-
-	-	B	-	-	A	A	D	-	-	D	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
D	B	B	-	-	A	A	A	D	-	D	-	-	C	D	-	-	-	-	-	-	-	-	-	-	-	-

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

MATERIAL COMPATIBILITY TABLE (continued)	METALS											PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester
Monoethanolamine	A	-	A	A	B	D	-	A	-	-	-	B	-	D	-	D	-	A	A	D	-
Morpholine	B	-	-	A	A	-	B	-	B	A	-	-	C	-	-	-	-	A	A	D	-
Motor oil	A	A	A	A	A	-	-	A	-	-	-	A	C	B	-	B	A	-	A	A	-
Mustard	C	B	A	A	B	A	A	A	A	A	-	A	B	C	-	A	A	A	A	A	-
Naphtha	B	B	A	A	A	A	A	A	B	B	-	B	C	B	-	A	A	A	A	B	-
Naphthalene	A	A	A	A	B	-	B	A	B	A	-	A	D	A	-	D	D	A	A	-	A
Natural Gas	A	B	A	A	A	-	A	A	D	-	-	-	B	B	-	A	-	-	A	-	-
Neon	A	A	A	A	A	A	A	-	A	-	-	-	-	-	-	-	-	-	-	-	-
Nickel Chloride	D	D	A	C	D	-	D	B	B	B	A	A	A	A	-	A	A	A	A	A	A
Nickel Nitrate	C	D	B	B	D	-	D	A	B	B	A	-	A	-	-	A	A	A	A	D	A
Nickel Sulfate	D	D	A	B	D	-	D	B	B	B	A	B	B	A	-	A	A	A	A	B	A
Nitric Acid 10%	D	D	A	B	A	D	D	A	D	A	-	A	B	D	C	A	A	A	D	B	D
Nitric Acid 35%	D	D	A	C	D	D	D	A	D	A	-	A	B	D	D	A	A	B	D	C	D
Nitric Acid 70%	D	D	B	D	D	D	D	A	D	A	-	A	C	D	D	D	B	C	D	C	D
Nitric acid, conc.	D	D	D	D	D	D	D	A	D	B	-	B	D	D	D	D	D	D	D	D	D
Nitric acid, fuming	D	D	D	C	-	-	-	-	-	A	-	C	-	-	-	-	-	-	D	-	D
Nitric Oxide	-	A	A	A	A	D	D	-	D	-	-	-	-	-	-	-	-	-	-	-	-
Nitrobenzene	C	B	A	A	B	B	D	A	B	D	-	A	D	C	-	D	D	C	C	D	D
Nitrogen	A	A	A	A	A	A	A	A	A	A	-	A	A	A	-	A	A	A	A	A	-
Nitromethane	-	-	A	A	A	A	-	-	-	-	-	-	D	A	-	D	-	A	B	D	-
Nitrous Acid 10%	D	D	A	-	D	C	D	B	D	D	-	-	D	-	-	A	A	D	-	-	-
Nitrous Oxide	C	B	A	A	B	B	B	D	D	B	A	-	-	-	-	A	-	-	C	-	-
Octane	-	-	A	A	B	B	B	B	-	A	-	-	-	-	-	A	-	A	-	-	-
Octanol 1-	A	A	A	A	B	B	-	A	-	A	-	B	-	A	-	-	B	A	A	-	-
Oils (animal)	A	A	A	A	-	-	A	-	A	-	-	-	-	-	-	C	-	-	A	-	-
Oils (lubricating, petroleum)	A	A	A	A	A	A	A	A	B	A	-	-	-	B	-	A	A	A	A	-	-
Oils (mineral)	B	B	A	A	A	B	A	A	A	A	-	A	A	A	C	B	A	A	A	C	-
Oils (silicone)	B	-	A	A	A	B	A	-	A	-	-	-	A	A	B	B	A	A	A	B	-
Oils (vegetable)	A	B	A	A	A	B	B	-	B	A	-	-	B	A	-	C	-	A	A	-	-
Oleic Acid	C	C	A	A	A	A	B	B	B	A	-	B	D	A	B	D	A	A	A	B	A
Oxalic Acid	C	D	B	C	A	B	D	B	B	B	-	A	A	B	-	A	A	A	D	-	-
Oxygen	B	B	A	A	A	A	A	-	A	-	-	-	A	A	-	A	A	-	A	-	-
Ozone	A	A	B	A	B	A	B	B	A	-	-	-	B	C	-	A	A	-	D	C	-
Paraffins	B	B	A	A	A	B	A	A	A	B	-	A	A	A	-	D	A	A	A	A	-
Paraformaldehyde	B	B	-	B	-	-	B	-	B	-	-	-	-	-	-	-	-	-	-	-	-
Pentane n-	B	B	A	A	B	-	A	C	B	A	-	-	D	B	-	A	-	A	A	A	-
Pentanol 1-	-	-	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Perchloric Acid 67%	D	D	C	C	D	D	-	B	-	B	-	D	-	C	B	B	A	-	D	-	-
Perchloroethylene	A	B	B	A	C	B	C	B	B	B	-	A	D	B	-	C	C	D	C	D	-
Petrolatum (Petr. Jelly)	C	C	A	A	-	-	B	A	A	A	-	-	-	B	-	B	-	A	C	-	-
Petroleum Ether (naphtha)	B	B	A	A	A	A	A	-	B	-	B	-	C	B	-	A	A	A	B	-	-
Phenol 10%	D	D	B	B	A	B	-	B	-	B	-	B	D	B	D	A	A	C	D	D	-
Phenol 91%	D	D	B	B	A	D	D	B	-	A	-	A	D	D	D	C	B	C	D	D	-
Phosphine	-	A	A	A	A	D	-	-	A	-	-	-	-	-	-	-	-	-	-	-	-
Phosphoric Acid 10%	D	D	B	A	C	D	D	-	D	A	-	B	B	D	B	A	A	A	D	C	-
Phosphoric Acid 25%	D	D	B	B	C	D	D	B	C	A	-	C	B	D	B	A	A	A	D	C	-
Phosphoric Acid 85%	D	D	C	C	C	D	D	B	B	A	-	C	C	D	C	A	A	B	D	D	A
Phosphorous Oxychloride	D	D	D	D	B	D	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Phosphorous Trichloride	C	B	A	A	D	D	-	-	-	A	A	A	D	D	-	A	D	A	-	C	-
Phthalic Acid	C	C	B	A	B	C	B	B	A	B	-	A	B	C	-	-	B	-	B	-	-
Phthalic Anhydride	C	C	A	A	A	C	B	A	A	A	-	-	B	C	-	-	D	-	A	A	-
Picric Acid	B	D	B	B	C	D	C	B	D	B	-	A	A	A	-	A	D	A	B	D	-
Piperidine	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-	-	-	-
Polyvinyl acetate	B	-	-	B	-	-	B	-	B	-	-	-	-	-	-	-	-	-	-	-	-
Potassium Bicarbonate	A	-	A	B	D	B	-	B	-	B	-	A	A	C	D	A	A	A	A	-	D
Potassium Bisulfate	-	-	-	C	-	-	-	-	B	-	-	-	-	-	-	-	-	-	-	-	-
Potassium Bromide	D	D	B	B	C	B	C	B	B	B	A	A	A	A	-	A	A	A	C	A	-
Potassium Carbonate	C	B	A	B	D	B	B	B	B	B	A	A	A	B	-	A	A	A	A	-	A
Potassium Chlorate	C	B	A	A	B	B	B	B	C	B	A	B	A	B	-	A	A	A	D	A	-
Potassium Chloride	A	B	A	A	D	B	D	B	B	B	A	A	A	A	-	A	A	A	A	A	A

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

MATERIAL COMPATIBILITY TABLE (continued)	METALS												PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester	
Potassium Chromate	A	-	B	B	B	-	B	B	B	A	-	-	-	C	-	A	A	C	-	-	-	
Potassium Cyanide	B	B	A	B	D	D	D	D	B	B	A	A	A	C	-	A	A	C	A	D	A	
Potassium Dichromate	B	C	B	B	B	B	D	B	B	B	A	A	B	B	-	A	A	A	B	-	-	
Potassium Ferricyanide	C	C	B	B	B	B	D	B	B	B	A	A	B	B	-	A	A	A	B	A	-	
Potassium Ferrocyanide	C	C	B	B	B	B	B	B	A	B	A	A	-	-	-	A	A	A	A	A	-	
Potassium Hydroxide >50%	B	B	B	B	D	B	D	D	A	B	D	D	A	A	D	A	A	A	A	D	D	
Potassium Hydroxide 10%	A	A	B	A	-	-	D	-	A	-	B	-	A	B	D	A	-	-	A	D	-	
Potassium Hypochlorite	A	-	D	D	D	D	-	D	-	B	-	A	-	-	-	A	-	-	B	-	-	
Potassium Iodide	B	C	A	A	B	A	D	A	C	A	A	A	B	-	-	A	A	-	A	-	-	
Potassium Nitrate	A	B	A	B	B	A	B	B	B	B	A	B	B	A	-	A	A	A	C	A	A	
Potassium Oxalate	A	-	B	B	B	B	-	A	-	A	A	A	-	-	-	-	-	-	-	-	-	
Potassium Permanganate	B	B	A	B	B	A	B	A	B	A	A	B	B	A	-	A	A	A	D	A	A	
Potassium Sulfate	B	B	A	B	C	B	C	A	B	B	A	A	B	B	-	A	A	A	C	A	-	
Propane gas	B	B	A	A	A	A	A	A	B	A	-	-	-	A	-	A	A	A	A	C	-	
Propanol, 1-	A	A	-	A	A	B	A	A	A	A	-	B	D	A	B	A	A	B	A	B	-	
Propyl Acetate	A	A	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Propylene	A	-	B	A	A	A	-	-	-	-	-	-	B	-	-	B	-	-	-	-	-	
Propylene Glycol	B	B	B	B	B	A	B	A	B	B	-	A	B	B	-	A	C	B	B	B	-	
Propylene Glycol Methyl Ether	-	-	-	-	-	-	-	-	-	-	-	-	-	-	D	-	-	C	-	-	D	
Propylene Oxide	B	B	A	A	B	B	-	-	-	-	-	-	-	-	-	A	-	-	-	-	-	
p-Toluenesulfonic acid	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Pyridine	A	A	C	A	B	B	B	B	-	B	-	B	-	B	-	D	D	A	-	D	-	
Pyrrole	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	
Pyrogalllic Acid	D	B	B	B	B	B	-	A	-	B	-	A	-	D	-	B	A	A	A	-	-	
Resorcinol	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	-	-	-	D	B	-	
Rust Inhibitors	C	-	A	A	-	-	-	A	-	-	-	-	-	A	-	-	-	A	A	-	-	
Salicylic Acid	C	D	B	B	B	A	C	A	B	A	-	A	A	D	-	B	-	-	A	A	-	
Salt Brine (NaCl, saturated)	D	C	B	A	B	B	B	B	B	A	-	A	-	-	-	A	A	A	-	A	-	
Sea Water	D	D	C	B	B	B	D	A	A	A	-	A	-	A	-	A	A	A	A	A	-	
Silane	-	A	A	A	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	-	-	
Silver Bromide	D	-	D	D	D	-	-	D	B	A	A	-	-	C	-	-	-	A	-	-	-	
Silver Nitrate	C	D	A	B	D	-	D	B	D	A	A	A	B	A	-	A	A	A	A	B	A	
Soap Solutions	A	A	A	A	C	A	B	B	A	A	-	A	A	A	-	A	A	A	A	A	-	
Sodium Acetate	B	C	A	B	B	A	B	B	B	A	A	A	B	B	-	A	A	A	A	A	A	
Sodium Aluminate	B	C	A	A	-	-	B	A	B	B	-	A	-	B	-	-	-	A	A	-	-	
Sodium Benzoate	-	-	-	A	A	-	-	A	B	A	A	A	A	-	-	A	A	A	-	A	-	
Sodium Bicarbonate	C	C	A	B	D	B	C	A	B	B	A	A	A	A	D	A	A	A	A	A	A	
Sodium Bisulfate	D	D	A	B	D	B	C	A	B	B	A	A	A	B	-	A	A	A	C	A	A	
Sodium Bisulfite	D	D	A	B	D	B	B	B	B	B	-	A	A	C	D	A	A	A	D	B	-	
Sodium Borate (Borax)	C	C	B	B	C	B	B	A	B	A	A	B	A	-	-	C	A	A	A	A	-	
Sodium Bromide	C	C	C	C	D	D	B	A	B	-	A	A	B	A	-	B	A	A	B	-	-	
Sodium Carbonate	B	B	A	A	D	A	B	A	B	A	A	A	B	A	-	A	A	C	A	A	-	
Sodium Chlorate	C	C	A	B	C	B	B	B	B	B	A	A	A	A	-	A	A	A	A	A	D	
Sodium Chloride	C	C	A	C	C	B	C	B	A	A	A	A	A	A	B	A	A	A	A	B	A	
Sodium Chromate	A	B	B	B	B	B	C	B	B	A	-	-	-	D	-	-	-	C	A	A	-	
Sodium Cyanide	A	B	A	B	D	D	D	D	B	A	-	A	A	A	-	A	A	A	C	-	A	
Sodium Ferrocyanide	-	-	B	B	A	D	-	-	B	A	A	-	-	A	-	-	A	A	-	-	-	
Sodium Fluoride	C	D	D	D	B	D	C	A	B	A	D	A	A	-	-	A	A	A	A	-	-	
Sodium Hydrosulfite	-	-	-	-	A	-	-	-	-	A	A	-	-	-	-	C	C	-	A	-	-	
Sodium Hydroxide >50%	A	A	A	B	D	B	A	C	A	C	D	A	A	A	B	A	A	A	C	D	-	
Sodium Hydroxide 15%	A	A	A	A	D	A	A	B	A	B	C	A	B	A	B	A	A	A	C	C	-	
Sodium Hypochlorite 12.2%	D	D	C	D	D	-	D	C	D	A	-	A	B	D	C	A	A	C	A	B	D	
Sodium Hypochlorite 5.5%	D	D	C	D	D	-	D	-	D	B	-	-	B	-	C	A	A	C	A	B	D	
Sodium Metaphosphate	C	B	A	A	C	B	D	A	-	-	-	-	-	B	-	A	A	A	-	-	-	
Sodium Metasilicate	C	C	A	A	D	B	B	A	A	A	-	-	-	D	-	A	A	A	-	-	-	
Sodium Nitrate 3.5%	B	B	A	B	B	D	B	B	B	B	A	A	-	A	B	A	A	A	A	D	A	
Sodium Perborate	C	B	B	B	C	B	C	B	B	B	-	-	-	B	-	-	A	B	A	-	-	
Sodium Peroxide	C	C	A	A	C	B	D	A	B	B	C	-	-	D	-	A	A	C	B	A	-	
Sodium Polyphosphate	D	-	B	B	D	A	D	B	-	A	-	A	-	B	-	A	A	A	A	-	-	
Sodium Silicate	B	B	A	B	A	B	D	B	-	B	A	A	-	C	-	A	A	A	A	-	-	

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

MATERIAL COMPATIBILITY TABLE (concluded)	METALS											PLASTICS									
	Cast Iron	Carbon Steel	304 Stainless	316 Stainless	Aluminum	Copper	Brass	Bronze	Monel	Hastelloy C	Tantalum	Titanium	ABS	Acetal (Delrin)	Acrylic	PVC (Type 1)	CPVC	Epoxy	Nylon	Polycarbonate	Polyester
Sodium Phosphate	C	C	-	A	-	-	C	-	B	-	A	-	-	-	-	-	-	-	-	-	-
Sodium Sulfate	B	B	A	A	A	B	B	B	B	B	A	A	-	B	-	A	A	A	A	D	D
Sodium Sulfide	C	B	A	B	D	D	D	B	B	B	A	A	-	B	-	A	A	A	A	-	-
Sodium Sulfite	A	-	B	A	C	D	D	B	B	B	A	A	-	-	-	A	A	A	D	-	-
Sodium Tetraborate	A	-	A	A	C	B	-	A	A	-	-	-	-	B	-	A	A	A	-	-	-
Sodium Thiosulfate (hypo)	C	B	A	B	A	D	D	A	B	A	-	A	-	C	-	A	A	A	A	D	-
Stannic Chloride	D	D	D	D	D	-	-	A	-	B	A	A	-	C	-	A	A	A	A	A	A
Stannic Fluoborate	D	-	-	A	-	-	-	-	-	-	-	-	-	C	-	-	-	A	-	-	-
Stannous Chloride	-	D	D	C	D	-	-	A	-	B	A	A	-	-	-	A	A	A	D	-	A
Steam	A	A	-	A	D	-	A	-	B	-	-	B	-	-	-	-	-	-	-	-	-
Stearic Acid	C	C	B	A	B	D	D	B	B	B	-	A	-	A	-	A	B	B	A	A	-
Stoddard Solvent	A	B	A	A	A	A	-	A	-	A	-	A	B	A	-	A	C	A	A	A	-
Styrene Monomer	B	A	A	A	A	B	A	A	B	D	-	-	-	A	-	D	D	A	A	D	-
Sugar (Liquids)	B	B	A	A	A	A	A	A	A	A	A	-	B	A	-	-	-	A	A	-	-
Sulfur Chloride	D	D	D	D	D	B	C	B	B	A	A	D	-	D	-	D	C	C	A	-	-
Sulfur Dioxide gas (dry)	A	A	A	A	B	A	B	B	A	B	A	A	-	B	-	A	A	A	A	A	-
Sulfur Dioxide gas (wet)	-	A	A	A	B	B	B	B	A	C	A	A	D	B	-	A	A	A	D	-	-
Sulfur Hexafluoride	-	A	A	A	A	A	B	-	A	-	-	-	-	-	-	B	-	-	B	-	-
Sulfur Trioxide (dry)	A	B	D	B	A	B	A	B	B	B	-	D	-	D	-	A	A	A	D	-	-
Sulfur Trioxide (wet)	B	-	A	C	A	-	D	C	-	-	-	-	-	-	-	B	A	A	D	-	-
Sulfuric Acid 10%	C	D	D	C	D	D	C	B	B	B	-	D	B	D	C	A	A	A	D	B	A
Sulfuric Acid 30%	D	D	D	D	D	D	C	B	B	B	-	D	B	D	D	A	A	A	D	C	A
Sulfuric Acid 98%	D	A	D	D	D	D	C	B	B	B	-	D	-	D	D	D	C	C	D	D	C
Sulfuric acid, fuming	D	A	D	D	D	-	D	B	-	D	-	D	-	D	-	-	D	D	D	D	-
Sulfurous Acid	D	D	C	B	B	D	D	B	D	B	-	A	-	C	-	A	A	A	D	-	-
Sulfuryl Chloride	-	-	-	-	-	-	-	-	-	-	-	-	-	A	-	A	-	A	-	-	-
Tannic Acid	C	C	A	A	C	A	B	B	B	B	-	A	-	B	B	B	A	A	D	C	A
Tartaric Acid	C	D	A	C	B	A	C	B	B	B	-	A	-	B	-	A	A	A	A	-	A
Tetrachloroethane	A	-	B	A	C	A	-	-	-	A	-	A	-	A	-	D	C	A	B	-	-
Tetrachloroethylene	A	-	A	A	-	A	-	-	-	B	-	-	-	A	-	D	D	-	-	D	-
Tetraethylene Glycol	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
Tetrahydrofuran	-	A	A	A	-	-	-	A	-	A	-	B	-	A	-	D	D	A	A	D	-
Therminol D-12 HTF	A	A	A	A	A	A	A	A	-	-	-	-	A	A	D	A	-	A	A	A	C
Thionyl Chloride	D	D	-	-	-	-	-	-	-	-	-	-	-	-	-	A	-	-	-	-	-
Toluene	A	A	A	A	A	A	A	A	A	A	-	A	D	C	D	D	D	B	A	D	B
Trichlorobenzene	A	A	A	A	A	-	-	-	-	-	-	-	-	-	-	D	-	-	-	-	-
Trichloroacetic Acid 90%	D	-	D	D	D	D	B	-	B	B	-	D	-	-	-	-	-	D	C	D	-
Trichloroethane 1,1,1-	B	-	C	A	D	-	-	A	-	A	-	A	-	A	-	D	-	A	-	D	-
Trichloroethylene	C	B	B	B	D	A	B	B	B	A	-	A	D	D	D	D	D	C	C	D	-
Trichloropropane	A	-	A	A	D	A	-	A	-	A	-	-	D	A	-	D	-	A	-	-	-
Tricresyl Phosphate	B	-	B	B	D	B	-	A	-	A	-	B	B	C	-	C	D	A	A	-	-
Triethanolamine	-	B	A	A	-	B	-	-	B	A	A	A	-	-	-	D	-	B	A	-	-
Triethylamine	A	-	A	A	-	A	B	-	-	-	-	-	-	D	-	A	A	A	A	-	-
Triethylene Glycol	-	-	-	A	-	-	-	-	-	A	-	-	-	-	-	-	-	-	-	-	-
Trisodium Phosphate	-	-	B	A	D	B	-	A	-	A	-	-	B	A	-	A	A	A	A	-	D
Turpentine	B	B	A	A	A	B	C	A	B	B	-	B	D	A	D	D	A	B	A	D	-
Urea	C	C	A	A	B	-	B	B	B	B	-	A	B	A	-	A	A	-	A	D	-
Uric Acid	D	-	B	B	D	A	-	B	-	B	-	A	-	-	-	A	-	-	A	-	-
Vinegar	D	D	A	A	D	B	D	A	B	A	-	A	A	B	-	A	A	A	A	-	-
Vinyl Acetate	B	-	B	B	A	B	B	-	B	-	-	-	-	-	-	D	D	A	-	-	-
Vinyl Chloride	B	-	B	A	B	B	-	A	-	A	-	A	D	-	-	D	D	-	A	-	-
Water	D	D	A	A	B	B	B	A	A	A	A	A	A	A	-	A	A	A	A	A	A
Water, Deionized	D	D	A	A	A	B	A	-	-	A	A	A	-	-	-	A	A	A	A	-	-
Water, Distilled	D	D	A	A	A	B	A	A	A	A	A	A	B	B	-	A	A	A	A	A	-
Wine	D	D	A	A	C	B	B	A	A	A	-	A	C	A	-	A	A	B	A	-	-
Xylenes	B	B	A	A	A	A	A	A	A	A	A	A	D	A	D	D	D	A	A	D	B
Zinc Chloride	D	D	D	B	D	C	D	B	-	B	A	A	A	C	B	A	A	A	A	B	A
Zinc Hydrosulfite	D	-	A	A	D	B	-	-	-	-	-	-	A	C	-	-	-	A	A	-	-
Zinc Sulfate	D	D	A	A	D	B	B	B	B	A	A	A	A	C	B	C	A	A	A	B	A

A - Excellent

B - Good

C - Fair

D - Unacceptable

See notes at beginning of table.

A - Excellent B - Good C - Fair D - Unacceptable See notes at beginning of table.

Substances Known to Attack 316 Stainless Steel

Substance	Concentration %	Temp. °C	Substance	Concentration %	Temp °C
Aluminum Chloride	10	20	Hydrochloric Acid	0.5	50
Anilinium HCl	5	20	Hydrochloric Acid	2	20
Aqua Regia	-	20	Hydrofluoric	10	20
Caustic Potash	molten	360	Nitric Acid	65	boiling
Caustic Soda	molten	318	Nitric Acid	99	20
Chlorosulfonic Acid	10	20	Oxalic Acid	10	boiling
Chromium Trioxide	50	boiling	Phosphoric Acid	80	boiling
Cupric Chloride	1	75	Potassium Bisulfate	2	90
Cupric Chloride	sat.	20	Sodium Chlorite	5	20
Ferric Chloride	10	20	Stannic Chloride	aq.	boiling
Formic Acid	80	boiling	Stannous Chloride	sat.	boiling
HCl gas	dry	100	Sulfuric Acid	10	70
HNO ₃ /H ₂ SO ₄ mix	5/30	boiling	Sulfuric Acid	2.5	boiling
HNO ₃ /H ₂ SO ₄ mix	50/50	20	Tartaric Acid	25	boiling
Hydrazinium Sulfate	10	boiling	Trichloroacetic Acid	any	20

Substances Known to Attack Hastelloy C-276

Substance	Concentration %	Temp. °C	Substance	Concentration %	Temp °C
Fluorinating agents (i.e. Ant. Fluorochloride)	any	20	Hydrochloric Acid + 2% HF	5	70
Aqua Regia	-	20	P ₂ O ₅	44	116
Chromic Acid	-	20	P ₂ O ₅ + 0.5% HF	38	85
Cupric Chloride / HCl mix	any*	20	Nitric Acid	65	Boiling
Ferric Chloride / HCl mix	any*	20	Nitric Acid + 6% HF	5	60
HBr (aq.)	in presence of O ₂ or ox. cations (Fe-III)	>50	Nitric Acid + 25% H ₂ SO ₄ + 4% NaCl	5	Boiling
HCl (aq.)	"	>50	H ₂ SO ₄ / HCl	10/1	Boiling
Mineral Acids	"	>50	H ₂ SO ₄ / -Cl	10/200ppm	Boiling
Hydrochloric Acid	5	Boiling	H ₂ SO ₄ / Fe ₂ (SO ₄) ₃	10/4	Boiling
Hydrochloric Acid + 42g/l Fe ₂ (SO ₄) ₃	1	93	Ferric Chloride	6%	95

*attack made worse by polyamines and other complexing agents

Substances Known to Attack Tantalum Metal

Substance	Concentration %	Temp °C
Sodium Hydroxide	40	boiling
Hydrogen Peroxide	concentrated	all*
Sulfuric Acid	concentrated	250

*resistance to attack is generally fair

The tables above indicate some conditions known to cause corrosion of three materials of great importance in the CPI. Stainless steel is widely used for reaction vessels, piping and other components because of its mechanical strength and generally excellent resistance to chemical attack. However, as the table indicates, strong acids and a number of other mixtures should be avoided. Although corrosion rates are not given, it should be assumed that attack is severe enough to recommend against its use. Hastelloy, while costly, has long been considered the substitute for stainless steel when more aggressive reagents must be used and glass is not an option. However, certain agents must also be avoided. Tantalum is an extremely expensive but highly resistant metal, often used for temperature probes and other components in glass-lined vessels. Very few substances are known to attack it. Sources [66, 75, 117, 179, 226, 227].

11 Miscellaneous

Contents

Unit Conversion Factors	11-2
Comparison of Unit Systems	11-4
Mathematical and Physical Constants	11-5
Useful Mathematical Relationships	11-5
Exponents and Logarithms	11-5
Polynomials	11-5
Geometric Formulas	11-6
Trigonometry	11-7
Continuous Stirred Tank Dilution Effects (Feed and Bleed)	11-7
Calculations for Asymmetric Crystallizations	11-7
Temperature Conversion Diagram	11-8
Pressure Conversion Diagram	11-9
Vacuum Conversion Diagram	11-10
Useful Cold Mixtures for the Laboratory	11-11
Decimal Equivalents of Fractions of an Inch	11-11
Standard Sieve Specifications	11-12
Mill Grade Wire Cloth Specifications	11-12
Industry-Related Acronyms and Abbreviations	11-13

Commonly Used Unit Conversion Factors

LENGTH

$\frac{25.4 \text{ mm}}{\text{in}}$	$\frac{2.54 \text{ cm}}{\text{in}}$	$\frac{12 \text{ in}}{\text{ft}}$	$\frac{39.37 \text{ in}}{\text{m}}$	$\frac{3.281 \text{ ft}}{\text{m}}$	$\frac{1.094 \text{ yd}}{\text{m}}$	$\frac{25.4 \text{ } \mu\text{m}}{0.001 \text{ in}}$
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AREA

$\frac{6.452 \text{ cm}^2}{\text{in}^2}$	$\frac{144 \text{ in}^2}{\text{ft}^2}$	$\frac{0.09290 \text{ m}^2}{\text{ft}^2}$	$\frac{10.76 \text{ ft}^2}{\text{m}^2}$	$\frac{9 \text{ ft}^2}{\text{m}^2}$
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VOLUME

$\frac{16.39 \text{ cm}^3}{\text{in}^3}$	$\frac{1728 \text{ in}^3}{\text{ft}^3}$	$\frac{35.31 \text{ ft}^3}{\text{m}^3}$	$\frac{61.025 \text{ in}^3}{\text{L}}$	$\frac{28.317 \text{ L}}{\text{ft}^3}$	$\frac{1000 \text{ L}}{\text{m}^3}$	$\frac{28.317 \text{ L}}{\text{ft}^3}$
$\frac{7.481 \text{ US Gal}}{\text{ft}^3}$	$\frac{3.785 \text{ L}}{\text{US Gal}}$	$\frac{264.17 \text{ US Gal}}{\text{m}^3}$	$\frac{1.201 \text{ US Gal}}{\text{UK Gal}}$	$\frac{219.96 \text{ UK Gal}}{\text{m}^3}$	$\frac{42 \text{ US Gal}}{\text{barrel (petr.)}}$	

MASS

$\frac{2.205 \text{ lbm}}{\text{kg}}$	$\frac{453.6 \text{ g}}{\text{lbm}}$	$\frac{0.4536 \text{ kg}}{\text{lbm}}$	$\frac{908 \text{ kg}}{\text{US Ton}}$	$\frac{2205 \text{ lbm}}{\text{Met. Ton}}$	$\frac{31.1 \text{ g}}{\text{oz. (troy)}}$	$\frac{28.45 \text{ g}}{\text{oz. (av.)}}$	$\frac{64.799 \text{ mg}}{\text{grain}}$
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FLOW

$\frac{0.00223 \text{ ft}^3/\text{sec}}{\text{US Gal/min}}$	$\frac{0.1337 \text{ ft}^3/\text{min}}{\text{US Gal/min}}$	$\frac{4.40 \text{ US Gal/min}}{\text{m}^3/\text{hr}}$	$\frac{0.264 \text{ US Gal/min}}{\text{Liter/min}}$	$\frac{1.699 \text{ m}^3/\text{hr}}{\text{ft}^3/\text{min}}$	$\frac{471.7 \text{ cm}^3/\text{sec}}{\text{ft}^3/\text{min}}$	$\frac{0.4719 \text{ L/sec}}{\text{ft}^3/\text{min}}$
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DENSITY

$\frac{8.35 \text{ lbm/US Gal}}{\text{g/cm}^3}$	$\frac{62.4 \text{ lbm/ft}^3}{\text{kg/L}}$	$\frac{0.00835 \text{ lbm/US Gal}}{\text{kg/m}^3}$	$\frac{0.0624 \text{ lbm/ft}^3}{\text{kg/m}^3}$	$\frac{7.481 \text{ lbm/ft}^3}{\text{lbm/US Gal}}$	$\frac{1 \text{ g/cm}^3}{\text{kg/L}}$	$\frac{1 \text{ g/L}}{\text{kg/m}^3}$
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CONCENTRATION

$\frac{119.84 \text{ g/L}}{\text{lbm/US Gal}}$	$\frac{7.481 \text{ lbm/ft}^3}{\text{lbm/US Gal}}$	$\frac{0.0624 \text{ lbm/ft}^3}{\text{g/L}}$	$\frac{62.4 \text{ lbm/ft}^3}{\text{kg/L}}$	$\frac{99.78 \text{ g/L}}{\text{lbm/UK Gal}}$	$\frac{6.229 \text{ lbm/ft}^3}{\text{lbm/UK Gal}}$
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PRESSURE

$\frac{760 \text{ mmHg}}{\text{atm}}$	$\frac{33.90 \text{ ft H}_2\text{O}}{\text{atm}}$	$\frac{406.8 \text{ in H}_2\text{O}}{\text{atm}}$	$\frac{10.333 \text{ kg/m}^2}{\text{atm}}$	$\frac{14.70 \text{ lb/in}^2}{\text{atm}}$	$\frac{29.92 \text{ in Hg}}{\text{atm}}$	$\frac{2116.8 \text{ lb/ft}^2}{\text{atm}}$
$\frac{1.0133 \text{ bar}}{\text{atm}}$	$\frac{1 \text{ torr}}{\text{mm Hg}}$	$\frac{13.596 \text{ kg/m}^2}{\text{mm Hg}}$	$\frac{10 \text{ torr}}{\text{cm Hg}}$	$\frac{136 \text{ kg/m}^2}{\text{cm Hg}}$	$\frac{345.3 \text{ kg/m}^2}{\text{in Hg}}$	$\frac{13.6 \text{ in H}_2\text{O}}{\text{in Hg}}$
						$\frac{14.223 \text{ lb/in}^2}{\text{kg/cm}^2}$
$\frac{2.307 \text{ ft H}_2\text{O}}{\text{lb/in}^2}$	$\frac{6892.9 \text{ Pa}}{\text{lb/in}^2}$	$\frac{51.7 \text{ mm Hg}}{\text{lb/in}^2}$	$\frac{0.0703 \text{ kg/cm}^2}{\text{lb/in}^2}$	$\frac{14.5 \text{ lb/in}^2}{\text{bar}}$	$\frac{1 \times 10^6 \text{ dyn/cm}^2}{\text{bar}}$	$\frac{100 \text{ kPa}}{\text{bar}}$
						$\frac{10 \text{ bar}}{\text{mPa}}$

ENERGY

$\frac{252.5 \text{ cal}}{\text{Btu}}$	$\frac{778.2 \text{ ft-lbf}}{\text{Btu}}$	$\frac{1055 \text{ J}}{\text{Btu}}$	$\frac{1.055 \text{ kJ}}{\text{Btu}}$	$\frac{4.184 \text{ J}}{\text{cal}}$	$\frac{1000 \text{ cal}}{\text{kcal}}$	$\frac{1.3558 \text{ J}}{\text{ft-lbf}}$	$\frac{1 \times 10^7 \text{ erg}}{\text{J}}$
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Historical definitions: Btu = energy to raise 1 lbm H₂O by 1°F kcal = energy to raise 1 kg H₂O by 1°C

POWER

$\frac{1 \text{ W}}{\text{J/sec}}$	$\frac{0.2931 \text{ W}}{\text{Btu/hr}}$	$\frac{3415 \text{ Btu/hr}}{\text{kW}}$	$\frac{56.92 \text{ Btu/min}}{\text{kW}}$	$\frac{1.341 \text{ HP}}{\text{kW}}$	$\frac{746 \text{ W}}{\text{electric HP}}$	$\frac{1.014 \text{ metric HP}}{\text{HP}}$
$\frac{0.7457 \text{ kW}}{\text{HP}}$	$\frac{2544 \text{ Btu/hr}}{\text{HP}}$	$\frac{42.44 \text{ Btu/min}}{\text{HP}}$	$\frac{12,000 \text{ Btu/hr}}{\text{ton (refrig.)}}$	$\frac{3.517 \text{ kW}}{\text{ton (refrig.)}}$	$\frac{13.14 \text{ HP}}{\text{boiler HP}}$	

HEAT TRANSFER COEFFICIENT

$\frac{5.678 \text{ W/m}^2\text{-K}}{\text{Btu/ft}^2\text{-hr}^\circ\text{F}}$	$\frac{11.622 \text{ W/m}^2\text{-K}}{\text{cal/hr-cm}^2\text{-}^\circ\text{C}}$	$\frac{1.162 \text{ W/m}^2\text{-K}}{\text{kcal/hr-m}^2\text{-}^\circ\text{C}}$	$\frac{2.047 \text{ Btu/ft}^2\text{-hr}^\circ\text{F}}{\text{cal/hr-cm}^2\text{-}^\circ\text{C}}$	$\frac{4.886 \text{ kcal/hr-m}^2\text{-}^\circ\text{C}}{\text{Btu/ft}^2\text{-hr}^\circ\text{F}}$
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THERMAL CONDUCTIVITY

$\frac{1.731 \text{ W/m-K}}{\text{Btu/hr-ft}^\circ\text{F}}$	$\frac{0.116 \text{ W/m-K}}{\text{cal/hr-cm}^\circ\text{C}}$	$\frac{6.230 \text{ kJ/m-K}}{\text{Btu/hr-ft}^\circ\text{F}}$	$\frac{14.892 \text{ cal/hr-cm}^\circ\text{C}}{\text{Btu/hr-ft}^\circ\text{F}}$	$\frac{3.600 \text{ kJ/m-K}}{\text{W/m-K}}$
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SPECIFIC HEAT

$\frac{4186.8 \text{ J/kg-K}}{\text{Btu/lbm}^\circ\text{F}}$	$\frac{4.187 \text{ kJ/kg-K}}{\text{Btu/lbm}^\circ\text{F}}$	$\frac{0.239 \text{ Btu/lbm}^\circ\text{F}}{\text{J/g-K}}$	$\frac{4.184 \text{ kJ/kg-K}}{\text{cal/g}^\circ\text{C}}$	$\frac{1.0007 \text{ cal/g}^\circ\text{C}}{\text{Btu/lbm}^\circ\text{F}}$
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ENTHALPY (CALORIC VALUE)

$\frac{2.326 \text{ kJ/kg}}{\text{Btu/lbm}}$	$\frac{0.430 \text{ Btu/lbm}}{\text{J/g}}$	$\frac{4.184 \text{ J/g}}{\text{cal/g}}$	$\frac{9.224 \text{ J/kg}}{\text{cal/lbm}}$	$\frac{252.2 \text{ cal/lbm}}{\text{Btu/lbm}}$	$\frac{0.556 \text{ cal/g}}{\text{Btu/lbm}}$
$\frac{278.7 \text{ kJ/m}^3}{\text{Btu/US Gal}}$	$\frac{232.08 \text{ kJ/m}^3}{\text{Btu/UK Gal}}$	$\frac{37.26 \text{ kJ/m}^3}{\text{Btu/ft}^3}$	$\frac{7.481 \text{ Btu/ft}^3}{\text{Btu/US Gal}}$	$\frac{4184 \text{ kJ/m}^3}{\text{cal/mL}}$	

VISCOSITY

$\frac{1 \text{ Pa-sec}}{\text{N-sec/m}^2}$	$\frac{100 \text{ cP}}{\text{poise}}$	$\frac{0.001 \text{ Pa-sec}}{\text{cP}}$	$\frac{1000 \text{ cP}}{\text{kg/m-sec}}$	$\frac{3.6 \text{ kg/m-hr}}{\text{cP}}$	$\frac{1.4882 \text{ Pa-sec}}{\text{lbm/ft-sec}}$	$\frac{1488.2 \text{ cP}}{\text{lbm/ft-sec}}$	$\frac{2.419 \text{ lbm/ft-hr}}{\text{cP}}$
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KINEMATIC VISCOSITY

$\text{cSt} = \frac{\text{Cp}}{\text{density}}$	$\frac{100 \text{ cSt}}{\text{stoke}}$	$\frac{25.807 \text{ cSt}}{\text{ft}^2\text{-hr}}$	$\frac{929 \text{ St}}{\text{ft}^2\text{-sec}}$	$\frac{277.8 \text{ cSt}}{\text{m}^2\text{-hr}}$	$\frac{3600 \text{ ft}^2\text{/hr}}{\text{ft}^2\text{/sec}}$	$\frac{1 \times 10^6 \text{ cSt}}{\text{m}^2\text{/sec}}$
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LINEAR VELOCITY / ACCELERATION

$\frac{0.3048 \text{ m/sec}}{\text{ft/sec}}$	$\frac{3.281 \text{ ft/sec}}{\text{m/sec}}$	$\frac{0.0051 \text{ m/sec}}{\text{ft/min}}$	$\frac{1.609 \text{ km/hr}}{\text{mi/hr}}$	$\frac{1.467 \text{ ft/sec}}{\text{mi/hr}}$	$\frac{0.3048 \text{ m/sec}^2}{\text{ft/sec}^2}$	$\frac{30.48 \text{ cm/sec}^2}{\text{ft/sec}^2}$
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Sources: [101, 154, 156, 176, 194, 195, 266]

Comparison of Unit Systems

There is a fundamental difference in the way that different unit systems are defined. The SI (Système International) and Absolute Metric systems are based on fundamental units of mass, length, time, and temperature. The British Gravitational system is based on fundamental units of force, length, time, and temperature. Under these systems, in Newton's second law of thermodynamics, $F = ma/g_c$, the proportionality constant g_c is dimensionless and has a value equal to unity. It can be safely ignored in engineering calculations. However, in the English Engineering system, the units of mass and force are both chosen as fundamental units. The proportionality constant is not dimensionless and is not equal to unity. Therefore, whenever calculations involving mechanical energy terms are performed in the English system, this factor must be used. If the proportionality constant is not included, results will be incorrect.

Some familiar equations are shown here to illustrate this:

	SI, Absolute and British Systems	English Engineering System
Force:	$F = ma$	$F = \frac{ma}{g_c}$
Potential Energy:	$PE = mgz$	$PE = \frac{mgz}{g_c}$
Kinetic Energy:	$KE = mv^2$	$KE = \frac{mv^2}{g_c}$

The table below summarizes some common fundamental and derived units used in various systems. Also shown at the bottom of the page are some common internationally used unit prefixes. Sources [169, 194, 207, 267].

Comparison of Systems of Units

Fundamental Units					Derived Units				
System	Unit of Mass	Unit of Length	Unit of Time	Unit of Temperature	Unit of Force	Unit of Energy	Unit of Heat	Unit of Power	Unit of Electrical Energy
SI (Système International)	kg	m	sec	K	$N = 1 \frac{\text{kg} \cdot \text{m}}{\text{sec}^2}$	$J = 1 \text{ N} \cdot \text{m}$	**Kcal = $4.1868 \times 10^3 \text{ J}$	$W = \text{J/sec}$	$\text{Kw/hr} = 3.6 \times 10^6 \text{ J}$
Absolute Metric	g	cm	sec	K	$\text{dyne} = 1 \frac{\text{g} \cdot \text{cm}}{\text{sec}^2}$	J	Kcal	W	Kw/hr
British Gravitational	*slug = $1 \frac{\text{lbf} \cdot \text{sec}^2}{\text{ft}}$	ft	sec	R	lbf	J	Kcal	W	Kw/hr
English Engineering	*lbm	ft	sec	R	lbf	Btu	Btu	HP = 2544 Btu/hr	Kw/hr

* In the British System, mass is a secondary unit, derived from the other fundamental units.

* lbm is related to lbf as: $1 \text{ lbf} = 1 \text{ lbm} \times g/g_c$ where $g = 32.17 \text{ ft/sec}^2$ and $g_c = 32.17 \text{ ft} \cdot \text{lbm/lbf} \cdot \text{sec}^2$.

** The International kcal = $4.1868 \times 10^3 \text{ J}$, whereas the thermochemical kcal is defined as $= 4.184 \times 10^3 \text{ J}$.

SI Unit Prefixes

Name	Value	Symbol
atto	10^{-18}	a
femto	10^{-15}	f
pico	10^{-12}	p
nano	10^{-9}	n
micro	10^{-6}	μ

Name	Value	Symbol
milli	10^{-3}	m
cent*	10^{-2}	c
deci*	10^{-1}	d
deca*	10	da
hecto*	10^2	h

Name	Value	Symbol
kilo	10^3	k
mega	10^6	M
giga	10^9	G
tera	10^{12}	T

* prefixes not recommended

Important Mathematical and Physical Constants

Constant	Value	Constant	Value
π	3.14159	N (Avogadro's No.)	6.02252×10^{23}
e	2.71828	Ideal gas molar volume at STP (1 atm, 0°C)	0.02241 m ³ /gmole
sqrt 2	1.4142		22.41 L/gmole
log e	0.4343		359 ft ³ /lbmole
ln 10	2.3026	Air density at STP	1.2929 g/L
R (ideal gas constant)	8.3145 J/mole-°K		0.08071 lbf·ft ³
	1.987 cal/gmole-°K	g_c (Gravitational constant)	6.6726×10^{-11} m ³ /kg-sec
	0.0821 l-atm/gmole-°K		32.17 ft-lbm/lbf-sec ²
	10.73 psi-ft ³ /lbmole-°R	g (Acceleration of gravity)	9.8067 m/sec ²
	1.987 Btu/lbmole-°R		32.18 ft/sec ²

Sources: [70, 121, 154, 195]

Useful Mathematical Relationships

Exponents and Logarithms

$$b^0 = 1$$

$$b^1 = b$$

$$b^{-n} = \frac{1}{b^n} = \left(\frac{1}{b}\right)^n$$

$$\left(\frac{a}{b}\right)^n = \frac{a^n}{b^n}$$

$$(ab)^n = a^n b^n$$

$$b^{m/n} = \sqrt[n]{b^m} = \left(\sqrt[n]{b}\right)^m$$

$$(b^n)^m = b^{nm}$$

$$b^m b^n = b^{m+n}$$

$$\frac{b^m}{b^n} = b^{m-n}$$

$$b^{1/n} = \sqrt[n]{b}$$

$$\sqrt[n]{ab} = \sqrt[n]{a} \sqrt[n]{b} = a^{1/n} b^{1/n} = (ab)^{1/n}$$

$$\log_b(b) = 1$$

$$\log_b(1) = 0$$

$$\log_b(b^n) = n$$

$$\log(x^a) = a \log(x)$$

$$\log(\sqrt[n]{x}) = \log(x^{1/n}) = \frac{\log(x)}{n}$$

$$\log(xy) = \log(x) + \log(y)$$

$$\log\left(\frac{x}{y}\right) = \log(x) - \log(y)$$

$$\log_a(x) = \log_b(x) \log_a(b)$$

$$\ln(x) = \sim 2.3026 \log_{10}(x)$$

$$e^{\ln(x)} = x$$

Polynomials

$$(a + b)(a - b) = a^2 - b^2$$

$$(a + b)^2 = a^2 + 2ab + b^2$$

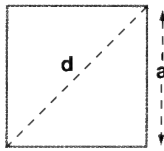
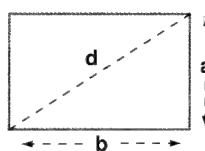
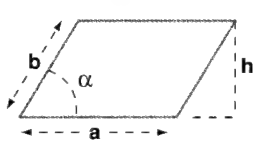
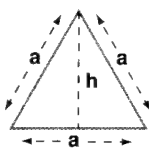
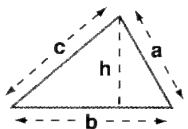
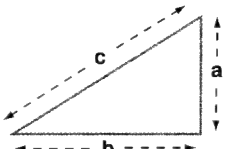
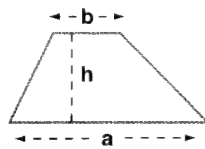
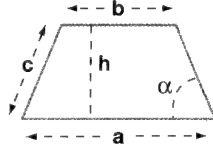
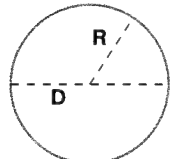
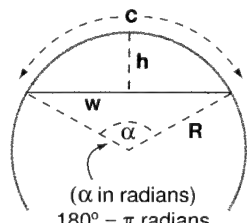
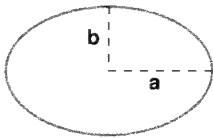
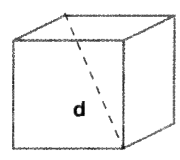
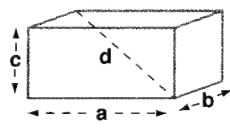
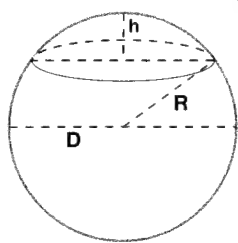
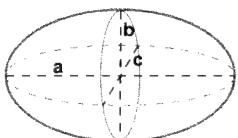
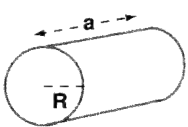
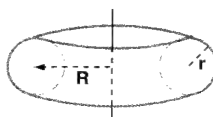
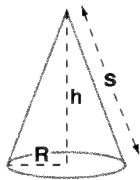
$$(a - b)^2 = a^2 - 2ab + b^2$$

$$(a + b)^3 = a^3 + 3a^2b + 3ab^2 + b^3$$

$$(a - b)^3 = a^3 - 3a^2b + 3ab^2 - b^3$$

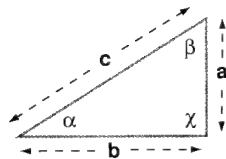
Sources: [154, 156, 195, 221]

Geometric Formulas

<p>SQUARE</p>  <p>Area = a^2 $d = a\sqrt{2}$</p>	<p>RECTANGLE</p>  <p>Area = ab $d = \sqrt{a^2 + b^2}$</p>	<p>PARALLELOGRAM</p>  <p>Area = $ah = absin\alpha$</p>	<p>EQUILATERAL TRIANGLE</p>  <p>Area = $\frac{1}{2}ah$ $h = \frac{1}{2}a\sqrt{3}$</p>
<p>OBLIQUE TRIANGLE</p>  <p>Area = $\frac{1}{2}bh$</p>	<p>RIGHT TRIANGLE</p>  <p>Area = $\frac{1}{2}ab$ $c = \sqrt{a^2 + b^2}$</p>	<p>TRAPEZOID</p>  <p>Area = $\frac{1}{2}h(a+b)$</p>	<p>ISOSCELES TRAPEZOID</p>  <p>Area = $\frac{1}{2}h(a+b)$ $= \frac{1}{2}csin\alpha(a+b)$</p>
<p>CIRCLE</p>  <p>Area = $\pi R^2 = \frac{1}{4}\pi D^2$ Circumference = πD</p>	<p>CIRCLE SEGMENT</p>  <p>Seg. Area = $\frac{1}{2}R^2(\alpha - sin\alpha)$ $\alpha = 2sin^{-1}(w/D)$ $h = R - \frac{1}{2}\sqrt{D^2 - w^2}$ $w = 2Rsin(\alpha/2)$ $c = \alpha R$ (α in radians) $180^\circ = \pi$ radians</p>		<p>ELIPSE</p>  <p>Area = πab</p>
<p>CUBE</p>  <p>Area = a^3 $d = a\sqrt{3}$</p>	<p>PARALLELOPIPED</p>  <p>Volume = abc $d = \sqrt{a^2 + b^2 + c^2}$ Total Surface = $2(ab+bc+ca)$</p>	<p>SPHERE</p>  <p>Area = $4\pi R^2 = \pi D^2$ Volume = $\frac{4}{3}\pi R^3$ Segment Cross-Sectional Area = $2\pi Rh$ Seg. Volume = $\frac{1}{3}\pi h^2(3R-h)$</p>	
<p>ELIPSOID</p>  <p>Volume = $\frac{4}{3}\pi abc$</p>	<p>CYLINDER</p>  <p>Area = $2\pi(R^2 + Ra)$ Volume = πR^2a</p>	<p>TORUS</p>  <p>Area = $4\pi^2 Rr$ Volume = $2\pi^2 Rr$</p>	<p>CONE</p>  <p>Volume = $\frac{1}{3}\pi R^2h$ Lateral Area = πRs</p>

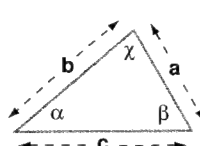
Sources: [70, 194, 221, 259]

Trigonometry



Right Triangle

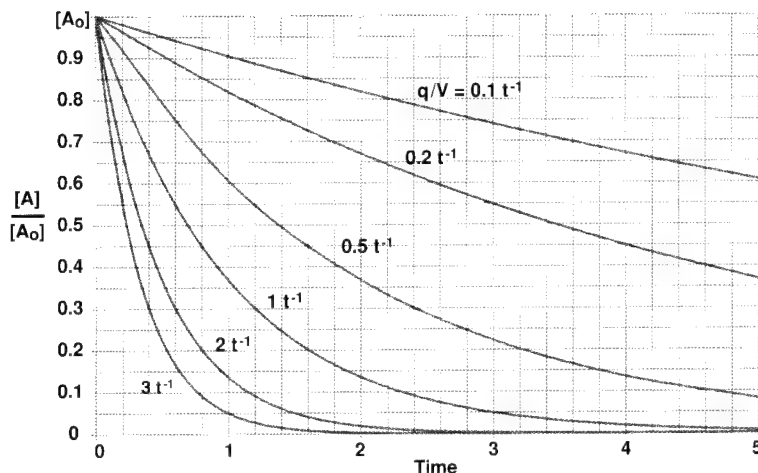
$$\begin{aligned}\sin \alpha &= a / c = \cos \beta \\ \cos \alpha &= b / c = \sin \beta \\ \tan \alpha &= a / b = \cot \beta \\ \cot \alpha &= 1 / \tan \alpha = b / a \\ \sec \alpha &= c / b \\ \operatorname{cosec} \alpha &= c / a \\ \sin \gamma &= 1 \quad \cos \gamma = 0\end{aligned}$$



Oblique Triangle

$$\begin{aligned}a &= \frac{b \sin \alpha}{\sin \beta} \quad b = \frac{a \sin \beta}{\sin \alpha} \quad c = \frac{a \sin \gamma}{\sin \alpha} \\ \sin \alpha &= \frac{a \sin \beta}{b} = \frac{a \sin \gamma}{c} \\ \cos \alpha &= \frac{b^2 + c^2 - a^2}{2bc}\end{aligned}$$

CST Dilution Effects (Feed and Bleed)



The formula for determining concentration vs. time for a Continuous Stirred Tank or other "feed and bleed" operation is shown below:

$$\ln \left(\frac{A}{A_0} \right) = \frac{q \cdot t}{V}$$

A_0 = starting concentration, A = concentration at time t , q = flowrate, and V = system volume. In this type of operation, volume remains constant while the flowrate in of fresh media ($[A] = 0$) equals the removal rate of reaction mixture. The concentration of A in the effluent is assumed to be equal to the concentration in the reactor (perfect mixing assumption). This relationship and the chart at left can be used in any time units as long as the units are used consistently throughout. Source [150].

Calculations for Asymmetric Crystallization

The following relationships are useful when performing asymmetric crystallizations of enantiomers and diastereomers, where ee = enantiomeric excess, $\%iso$ = percent isomeric purity, ee_0 = starting ee of the crystallization mixture, ee_{xII} = ee of the isolated crystals and ee_{mI} = ee of the crystallization mother liquors:

$$ee = (2 \times \%iso) - 1$$

$$\%iso = \frac{ee + 1}{2}$$

$$\text{Isomer Yield} = \text{Crystal Yield} \times (1 + ee_{xII})$$

When crystals and mother liquors are enriched in opposite isomers:

$$\text{Crystal Yield} = \frac{ee_0 + ee_{mI}}{ee_{xII} + ee_{mI}}$$

When crystals and mother liquors are enriched in the same isomer:

$$\text{Crystal Yield} = \frac{ee_0 - ee_{mI}}{ee_{xII} - ee_{mI}}$$

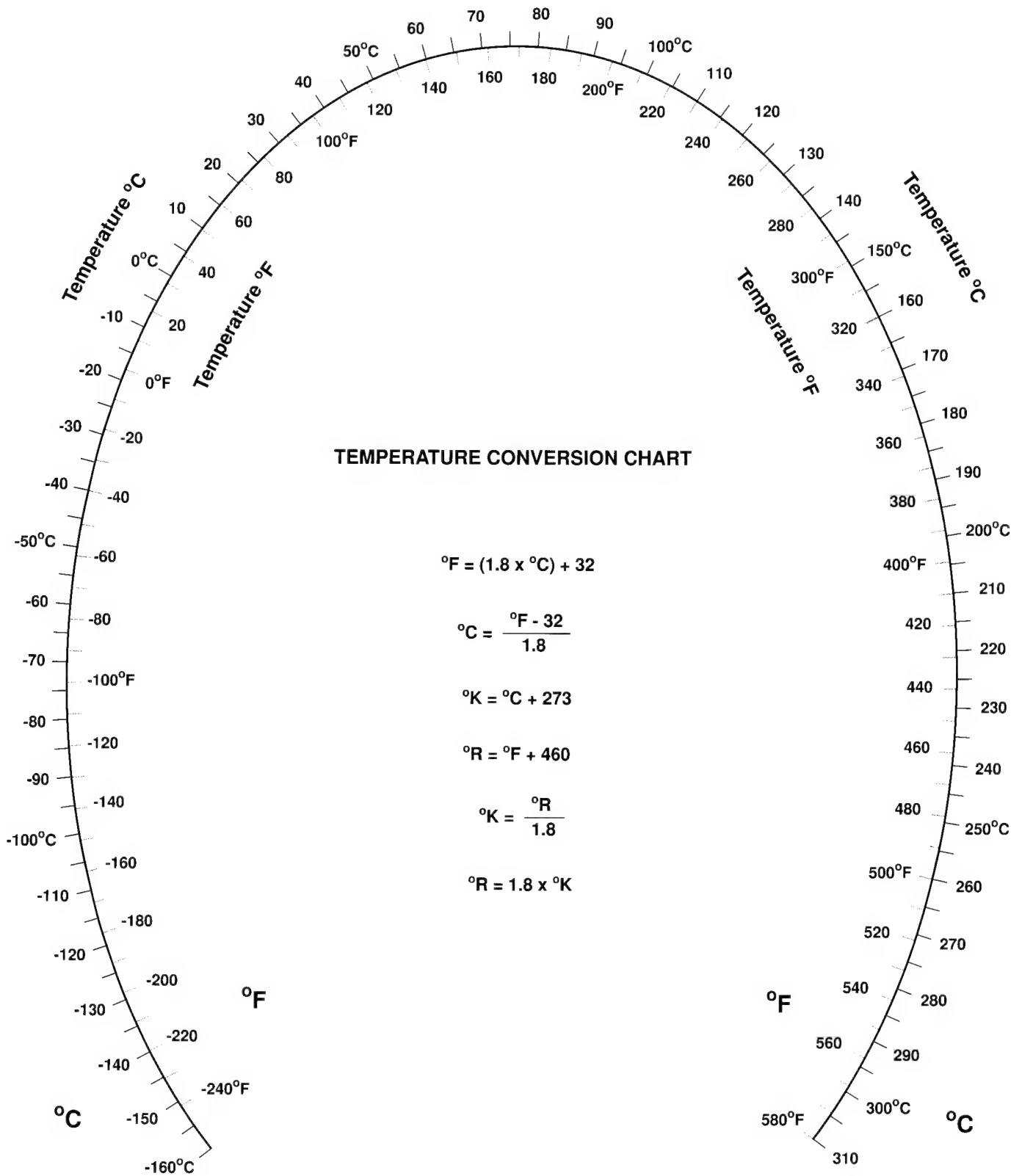
The solubility ratio (α) of two diastereomers is a measure of how good the resolving agent is (the higher the better). If the ee of the crystals is less than $\sim 90\%$, it is safe to assume that the solubility of both diastereomers has been exceeded. Therefore, the mother liquors are in equilibrium at their eutectic point and α can be estimated using:

$$\alpha = \frac{1 + ee_{mI}}{1 - ee_{mI}}$$

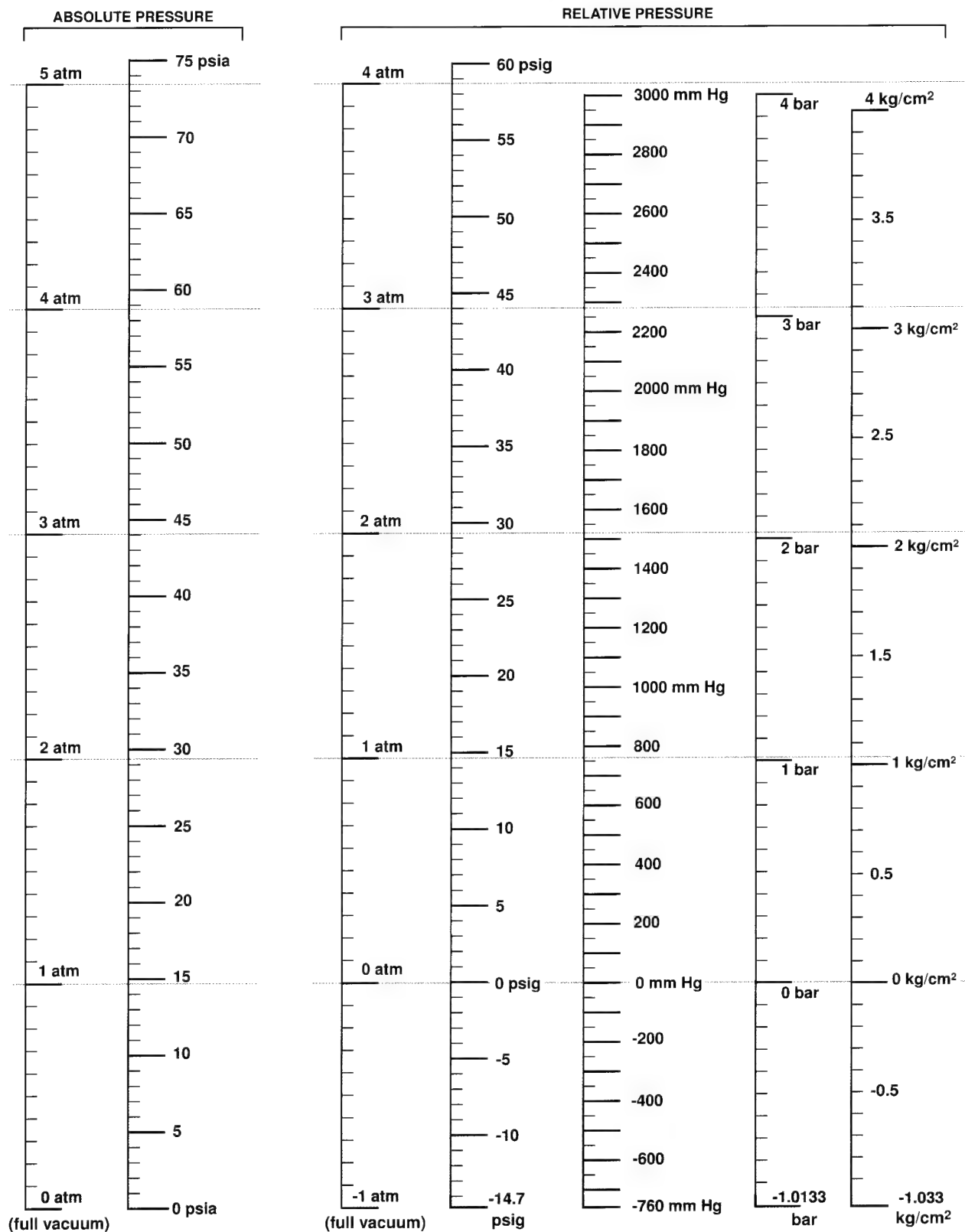
Then the highest possible yield of optically pure material which can be obtained can be estimated using:

$$\text{Maximum Yield} = 0.5 \times \left[1 - \frac{1}{\alpha} \right]$$

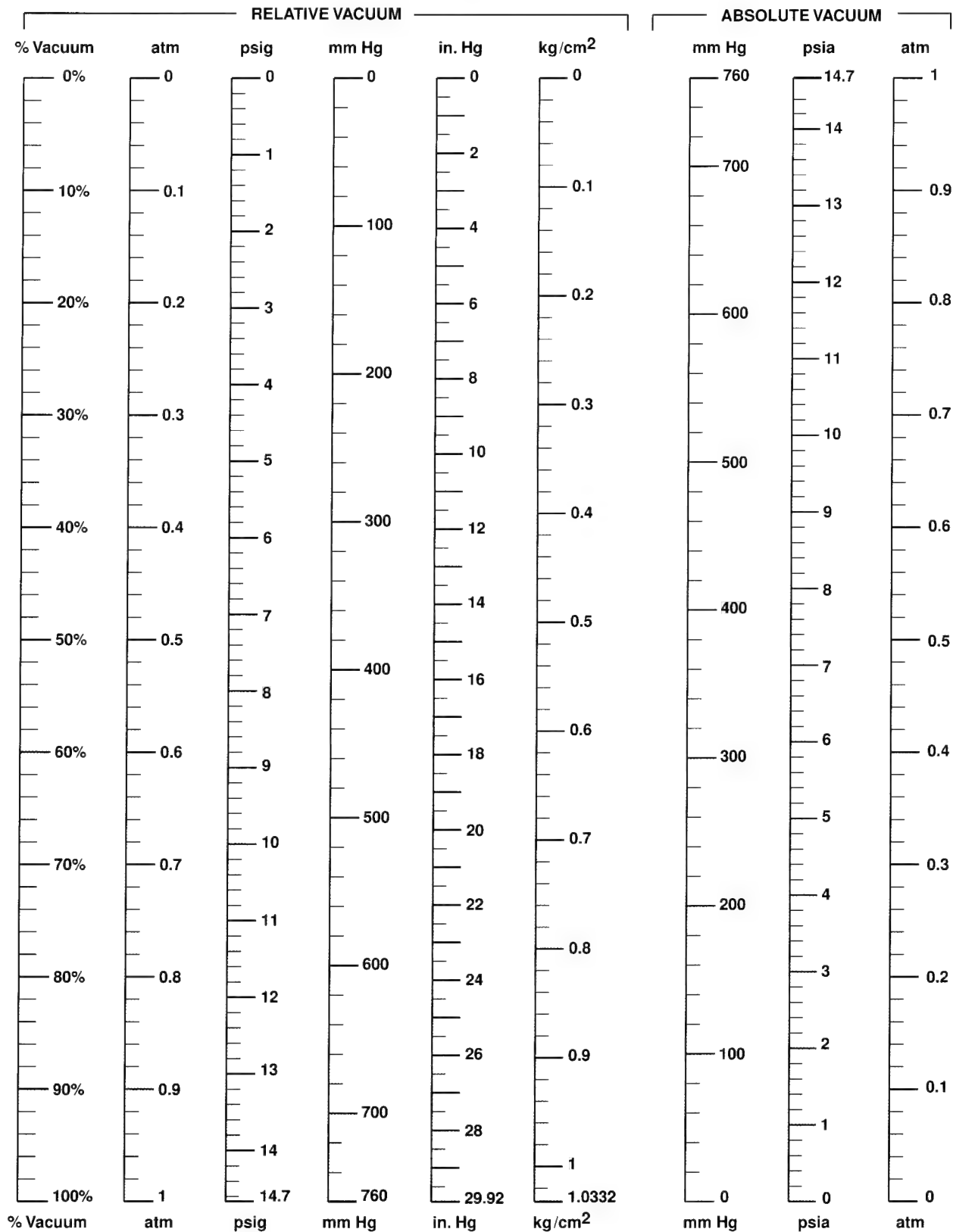
Source: J. Lopez [150].



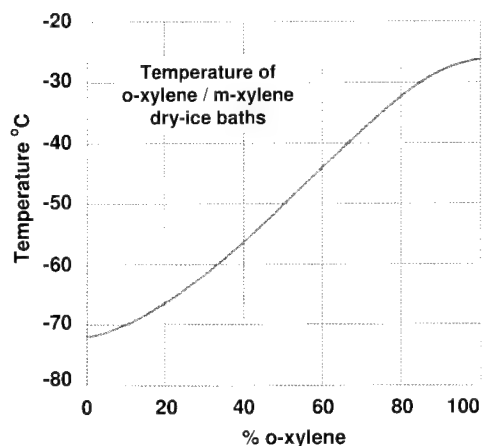
Pressure Conversion Diagram



Vacuum Conversion Diagram



Useful Cold Mixtures for the Laboratory



The table below lists some mixtures that can be used for making cold temperature baths in the laboratory or kilo-lab. These baths do not provide precise temperature control, and the flammability and toxicity of the substances listed must be taken into account before using them. In the case of the organic substances, once the desired temperature is established, the liquid nitrogen or dry ice (CO_2) is continuously added as necessary to maintain it. The inorganic salts are mixed with ice or water in the proportions indicated, and these baths can then be maintained by continually adding small amounts of finely divided dry ice as well. The chart at the left describes another useful method for achieving and maintaining fairly precise temperature by using a mixture of o-xylene and m-xylene with dry ice. The bath temperature is a function of the solvent mix composition. Sources [70, 110, 267].

Substance	Recipe	Approx. Temp. °C	Substance	Recipe	Approx. Temp. °C
p-xylene	liq. N_2 as needed	13	NaBr	66 g / 100 g ice	-28
Cyclohexane	liq. N_2 as needed	6	MgCl_2	85 g / 100 g ice	-34
NH_4NO_3	106 g / 100 g water	-4	$\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$	123 g / 100 g ice	-40
NH_4Cl	30 g / 100 g water	-5	Acetonitrile	dry ice as needed	-42
$\text{CaCl}_2 \cdot 6\text{H}_2\text{O}$	41 g / 100 g ice	-9	m-Xylene	liq. N_2 as needed	-47
KCl	30 g / 100 g ice	-11	n-Octane	liq. N_2 as needed	-56
Cycloheptane	liq. N_2 as needed	-12	Ethanol	dry ice as needed	-72
Ethylene Glycol	dry ice as needed	-15	Acetone	dry ice as needed	-86
NH_4Cl	25 g / 100 g ice	-15	Ethyl Acetate	liq. N_2 as needed	-84
NaNO_3	50 g / 100 g water	-18	Heptane	liq. N_2 as needed	-91
NaCl	33 g / 100 g ice	-21	Diethyl Ether	dry ice as needed	-100
Carbon Tetrachloride	dry ice as needed	-23	n-Pentane	liq. N_2 as needed	-131

The temperature of dry ice is normally -78.5°C . The atmospheric boiling point of liquid nitrogen is -196°C .

Decimal Equivalents of Fractions of an Inch

Fraction of an inch	Equivalent		Fraction of an inch	Equivalent		Fraction of an inch	Equivalent		Fraction of an inch	Equivalent	
	in.	mm		in.	mm		in.	mm		in.	mm
1/64	0.0156	0.3969	17/64	0.2656	6.7468	33/64	0.5156	13.0967	49/64	0.7656	19.4466
1/32	0.0313	0.7937	9/32	0.2813	7.1437	17/32	0.5313	13.4936	25/32	0.7813	19.8436
3/64	0.0469	1.1906	19/64	0.2969	7.5405	35/64	0.5469	13.8905	51/64	0.7969	20.2404
1/16	0.0625	1.5875	5/16	0.3125	7.9374	9/16	0.5625	14.2873	13/16	0.8125	20.6372
5/64	0.0781	1.9844	21/64	0.3281	8.3343	37/64	0.5781	14.6842	53/64	0.8281	21.0341
3/32	0.0938	2.3812	11/32	0.3438	8.7311	19/32	0.5938	15.0811	27/32	0.8438	21.4310
7/64	0.1094	2.7781	23/64	0.3594	9.1280	39/64	0.6094	15.4779	55/64	0.8594	21.8279
1/8	0.1250	3.1750	3/8	0.3750	9.5249	5/8	0.6250	15.8749	7/8	0.8750	22.2247
9/64	0.1406	3.5718	25/64	0.3906	9.9218	41/64	0.6406	16.2717	57/64	0.8906	22.6216
5/32	0.1563	3.9687	13/32	0.4063	10.3186	21/32	0.6563	16.6685	29/32	0.9063	23.0185
11/64	0.1719	4.3656	27/64	0.4219	10.7155	43/64	0.6719	17.0654	59/64	0.9219	23.4153
3/16	0.1875	4.7624	7/16	0.4375	11.1124	11/16	0.6875	17.4623	15/16	0.9375	23.8123
13/64	0.2031	5.1593	29/64	0.4531	11.5092	45/64	0.7031	17.8592	61/64	0.9531	24.2091
7/32	0.2188	5.5562	15/32	0.4688	11.9061	23/32	0.7188	18.2560	31/32	0.9688	24.6059
15/64	0.2344	5.9531	31/64	0.4844	12.3030	47/64	0.7344	18.6529	63/64	0.9844	25.0028
1/4	0.2500	6.3499	1/2	0.5000	12.6998	3/4	0.75	19.04976	1	1.0000	25.3997

Source: [49]

Standard Sieve Specifications

Sieve Designation	Nominal Opening in.	Nominal Opening μ m	Nominal Opening mm	Nominal Wire Diam in.	Nominal Wire Diam mm	Sieve Designation	Nominal Opening in.	Nominal Opening μ m	Nominal Opening mm	Nominal Wire Diam in.	Nominal Wire Diam mm
635	0.0008	20	0.02	0.0008	0.02	18	0.0394	1000	1	0.022	0.56
500	0.001	25	0.025	0.0009	0.02	16	0.0469	1190	1.19	0.025	0.64
450	0.0013	32	0.032	0.0009	0.02	14	0.0555	1410	1.41	0.028	0.71
400	0.0015	37	0.037	0.0011	0.03	12	0.0661	1680	1.68	0.031	0.79
325	0.0017	44	0.044	0.0013	0.03	10	0.0787	200	0.2	0.035	0.89
270	0.0021	53	0.053	0.0014	0.04	8	0.0937	2380	2.38	0.039	0.99
230	0.0025	63	0.063	0.0018	0.05	7	0.11	2790	2.79	0.044	1.12
200	0.0029	74	0.074	0.002	0.05	6	0.132	3350	3.35	0.049	1.24
170	0.0035	88	0.088	0.0024	0.06	5	0.157	4000	4	0.055	1.40
140	0.0041	105	0.105	0.0028	0.07	4	0.187	4760	4.76	0.063	1.60
120	0.0049	125	0.125	0.0035	0.09	3 1/2	0.223	5660	5.66	0.063	1.60
100	0.0059	149	0.149	0.0039	0.10	1/4 in	0.25	6350	6.35	0.07	1.78
80	0.007	177	0.177	0.005	0.13	0.265 in	0.265	6730	6.73	0.07	1.78
70	0.0083	210	0.21	0.0055	0.14	5/16 in	0.312	8000	8	0.079	2.01
60	0.0098	250	0.25	0.006	0.15	3/8 in	0.375	9510	9.51	0.088	2.24
50	0.0117	297	0.297	0.0078	0.20	7/16 in	0.438	11200	11.2	0.098	2.49
45	0.0139	354	0.354	0.0094	0.24	1/2 in	0.5	12700	12.7	0.098	2.49
40	0.0165	420	0.42	0.011	0.28	0.530 in	0.53	13500	13.5	0.11	2.79
35	0.0197	500	0.5	0.012	0.30	5/8 in	0.625	16000	16	0.124	3.15
30	0.0234	595	0.595	0.016	0.41	3/4 in	0.75	19000	19	0.124	3.15
25	0.0278	707	0.707	0.018	0.46	7/8 in	0.875	22200	22.2	0.14	3.56
20	0.0331	841	0.841	0.02	0.51	1 in	1	25400	25.4	0.14	3.56

Sieve sizes larger than 1/4 in. are designated by a number which equals the nominal opening size. Smaller sizes are designated by the number of openings per inch. By convention, when particle size is characterized by mesh designation, a plus sign or a minus sign is often used before the mesh size (i.e. +50 or -20), with + indicating that approximately 90% of the particles are retained by the sieve, and the - indicating that approximately 90% of the particles will pass through the sieve. Sources [8, 253].

Mill-Grade Wire Cloth Specifications

Mesh Count (per inch)	Wire Diam. (inches)	Mesh Opening (inches)	% Open Area	Mesh Count (per inch)	Wire Diam. (inches)	Mesh Opening (inches)	% Open Area
2 x 2	0.0540	0.1160	79.6	20 x 20	0.0140	0.0360	51.8
3 x 3	0.0410	0.2923	76.7	22 x 22	0.0135	0.0320	49.6
4 x 4	0.0350	0.2150	74.0	24 x 24	0.0130	0.0287	47.4
5 x 5	0.0320	0.1680	70.6	26 x 26	0.0110	0.0275	51.1
6 x 6	0.0280	0.1387	69.6	28 x 28	0.0100	0.0257	51.8
7 x 7	0.0280	0.1149	64.8	30 x 30	0.0095	0.0238	51.0
8 x 8	0.0250	1.0000	64.0	32 x 32	0.0090	0.0223	50.9
9 x 9	0.0230	0.0881	62.7	34 x 34	0.0090	0.0204	48.1
10 x 10	0.0200	0.0800	64.0	36 x 36	0.0090	0.0188	45.8
11 x 11	0.0200	0.0709	61.0	38 x 38	0.0085	0.0178	45.8
12 x 12	0.0180	0.0653	60.8	40 x 40	0.0085	0.0165	43.6
14 x 14	0.0170	0.0544	57.2	45 x 45	0.0080	0.0142	40.8
16 x 16	0.0160	0.0465	55.4	50 x 50	0.0075	0.0125	39.1
18 x 18	0.0150	0.0406	53.4	55 x 55	0.0070	0.0112	37.9

Wire cloth is also available in "Bolting Grades" which use finer wire and therefore have less tensile strength but more open area for a given comparable mesh size, and "Strainer Grades" which have non-symmetrical wire spacing and thus rectangular openings which generally provide for more open area and higher flows. Strainer Grade wire cloth is more easily formed than square mesh cloth. Most screens are available in a wide variety of materials. Sources [172, 228].

List of Industry-Related Acronyms and Abbreviations

ACGIH - American Conference of Government Industrial Hygienists
ACS - American Chemical Society
AIEE - American Institute of Electrical Engineers
AIHA - American Industrial Hygiene Association
ANSI - American National Standards Institute
API - American Petroleum Institute
ASME - American Society of Mechanical Engineers
ASTM - American Society for Testing and Materials
AWWA - American Water Works Association
BASEEFA - British Approvals Service for Electrical Equipment in Flammable Atmospheres
BOCA - Building Officials and Code Administrators International, Inc.
CDC - Centers for Disease Control
CENELEC - European Committee for Electrotechnical Standardization
CFR - Code of Federal Regulations
CGA - Compressed Gas Association
cGMP - Current Good Manufacturing Practice
CSA - Canadian Standards Association
DIN - Deutsches Institut für Normung (German Institute for Standardization)
DOT - Department of Transportation
EIA - Electronic Industries Alliance
EPA - Environmental Protection Agency
FDA - Food and Drug Administration
GMP - Good Manufacturing Practice
HAZWOPER - Hazardous Waste Operations and Emergency Response Standard
IAFIS - International Association of Food Industry Suppliers
IChemE - Institution of Chemical Engineers
IEC - International Electrotechnical Commission
IEEE - Institute of Electrical and Electronics Engineers, Inc.
ISO - International Organization for Standardization
IUPAC - International Union of Pure and Applied Chemists
MSDS - Material Safety Data Sheet
NEMA - National Electrical Manufacturers Association
NESC - National Electrical Safety Code
NFPA - National Fire Protection Association
NIOSH - National Institute of Occupational Safety and Health
NIST - National Institute of Standards and Technology
OSHA - Occupational Safety and Health Administration
PEI - Petroleum Equipment Institute
PMA - Pharmaceutical Manufacturers Association
RCRA - Resource Conservation and Recovery Act
RTECS - Registry of Toxic Effects of Chemical Substances (formerly Toxic Substances List)
SAE - Society of Automotive Engineers
TSCA - Toxic Substances Control Act
UL - Underwriters Laboratories, Inc.

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.....

.....

Index

3A sanitary standard 3-22
4-20 mA transmitter 5-17
ΔTLM 4-8
chart for estimating 4-10

A

Absolute pressure 5-18
Accumulation of reagents 2-13
Acetate buffer 8-8
Acid-base color indicators 8-9
Acid-base "flip-flop" 2-16
Acids
commercial, properties 8-3
concentration, commercial 8-3
dilution recipes 8-4, 8-5
multi-protic 8-8
pH of solutions 8-3
pKa in water 8-6
properties, commercial 8-3
resistance in reactors 2-4
Acronyms, agencies and standards 11-13
Across the line starters 5-13
Addition rate calculation for limiting reagents 2-13
Adiabatic calorimetry 1-9
Affinity laws for centrifugal pumps 3-4
Agency acronyms 11-13
Agitation, in vessels
(see also Mixing) 2-7
Air
density vs. temperature 7-14
flow through orifices 7-20
flow through pipes and tubes 7-19
physical properties 7-14
specific heat vs. temperature 7-14
therm. conduct. vs. temperature 7-14
viscosity vs. temperature 7-14
Air, compressed 7-16
typical usage rates 7-17
Air compressors 7-16
efficiency 7-17
Alkylation reactions 8-20
"All in and heat" operation 1-6
Alloys, metal 10-5
Alternating current 5-3
Amination reactions 8-20
Amino acids, pKa in water 8-7
Ammonia, refrigerant 4-27
Amperes vs. watts 5-3
Anchor impeller 2-8

Antoine equation 6-23
API density scale 8-17
ASI, Inc. 2-14
ASME pressure rating 2-6, 2-24
Asymetric crystallization formulae 11-7
Avagadro's number 11-5
Avoid, 12 things to 1-6
Azeotrope
boiling point prediction 6-25
composition vs. pressure 6-26, 6-27
definition 6-25
tables, binary 6-28
tables, water-containing ternary 6-40

B

Baffles 2-4, 2-10
Balling density scale 8-17
Bases
commercial, properties 8-3
density, commercial 8-3
dilution recipes 8-4, 8-5
molarity - weight % conversion 8-15
pH of solutions 8-3
properties, commercial 8-3
solution densities 8-15
Batch
analytical results 1-14
checklist 1-12
cooling profiles 4-3, 4-4
distillation 2-19, 2-20
heating profiles 4-3
log sheet 1-5, 1-14
maximizing the value 1-5
production record 1-14
record 1-5, 1-14
ticket 1-5, 1-14
Batch operation, advantages 1-4
Batch stirred-tank reactor 2-4
Baumé density scale 8-17
Benzene 6-4
BEP (Best efficiency point) 3-3
Binary azeotrope tables 6-28
Blend time 2-9
Blinding, filter cloth 2-26
Boiling point, reduced pressure 6-22, 6-23
Borohydrides 2-11
Brines, freezing point chart 4-24
British gravitational units 11-4
Brix density scale 8-17
Broadbent Co. 2-25

Buchi GlasUster 2-4
Buchi, reactor diagram 2-6
Buffers
NBS standardized 8-9
pKa in water 8-7
solution preparation 8-8
some useful 8-9

C

Calendering, filter cloth 2-27
Calibration, pH meter 5-24
Calorimetry
adiabatic 1-9
and scale-up 1-5, 1-8, 1-9
isothermal 1-9
Campaign report 1-15
Carbon black 2-18
removal 2-18
Carry-over, batch-to-batch 2-34
Cartridge, polish filter 2-18
CAS number 6-5
Cascade control 5-25
Catalytic hydrogenation 2-14
optimization of 2-14
safe operation of 2-14
Cavitation
corrosion 10-6
in pumps 3-3
Celite 2-17
CENELEC 5-9, 11-13
Centrifuge, product 2-25
operating tips 2-25
CFCs 4-27
CGA connections 7-6
dimensions and specifications 7-8
recommended torque values 7-7
cGMP 1-16
Charging raw materials 2-11
charging wand 2-11
ranges 1-4
Check valves 3-21
Checklist
batch 1-12
distillation 2-20
Chemical hygiene
chemical hygiene plan 9-3
importance in scale-up 1-3
Chemical inventory 9-3
Chemical nomenclature 8-19
Chemical purity, grades 8-18
Chemical reaction types 8-20

Index

Chemical resistance table 10-10
Chemical seals, for pressure gauges 5-19
Chemicals
 incompatible 9-9
 labeling requirements 9-8
 proper storage 9-2
 safe handling 9-2
Chillers 4-26
 capacity 4-27
 capacity, chart 4-28
 circulating pump size 4-27
 cooling air requirements 4-27
 cooling water requirements 4-27
 cooling water temperature chart 4-28
 efficiency 4-27
 horsepower 4-27
 schematic 4-26
 size requirements 4-27
Chilton equation for heat transfer 4-6
Chromatography, and scale-up 1-6
Circuit breakers 5-2
Clean Water Act 9-10
Cleaning
 criteria 2-34
 reactors and equipment 2-34
 solutions 2-34
 spray ball 2-34
 typical procedure 2-34
Cocurrent flow 4-8
Coefficient of expansion 6-5
Cogeim, 3V, Inc. 2-25
Cold mixtures 11-11
Color indicators, pH 8-9
Color removal 2-18
Compressed air systems 7-16
 air tank capacity 7-18
 equivalent cubic feet 7-18
 sizing 7-16
Compressed gases
 charging setup diagram 7-9
 common cylinder types 7-4
 estimating cylinder contents 7-3
 physical properties 7-11
 safe handling 7-2
 tips for using 7-2
 typical cylinder markings 7-4
Compression fittings 3-15
Compressors
 air 7-16
 in chillers 4-26
Computational flow dynamics 2-8
Concentration
 comparison of scales 8-17
 ppm vs. weight % 8-17
Concentration cell 10-6
Condensation reactions 8-20
Conjugate acid-base pair 8-8
Constant rate drying 2-30

Contamination, batch-to-batch 2-34
Continuous stirred tank formula 11-7
Control, reaction 2-13
 in scale up 1-4
Controlled addition 1-8, 2-11
 example 2-13
 limiting reagent 2-13
Controlled cooling profile 4-4
Conversion
 fractions of inch to decimal 11-11
 pressure, diagram 11-9
 temperature, diagram 11-8
 unit conversion factors 11-2
 vacuum, diagram 11-10
Cooling
 profile in crystallization 2-22
 rate and scale-up 1-8
 typical profiles 4-4
 vessel, example 4-2
Copper pipe 3-16
Corrosion
 conversion factors 10-7
 galvanic 10-6
 of glass by acids 10-9
 of glass by bases 10-9
 of Hastelloy 10-28
 of metals 10-6
 of stainless steels 10-28
 of tantalum 10-28
 resistance of various materials 10-6
 resistance table 10-10
 types 10-6
Countercurrent flow 4-8
Critical process parameters 1-8
Critical temperature 6-19
Crosible filter cloth 2-27
Cryogenic liquids
 physical properties 7-12
 special precautions 7-3
Crystal
 morphology 2-21
 polymorph 2-21
 size distribution 2-21
Crystallization
 asymmetric, calculations for 11-7
 bench studies 1-8, 2-21
 co-solvents 2-23
 cooling profile 2-22
 design 2-21
 driving force 2-22
 effect of water 2-23
 estimating yield, example 2-22
 evaporative 2-23
 kinetic 2-22
 mixing effects 2-23
 plotting solubility data 2-22
 rind formation 2-23
 seeding 2-23
 single solvent 2-21

 solvents for 6-4
 tips for optimizing 2-23
CSTR calculations 11-7
Cuno 2-18
Current draw 5-3
 motors 5-12
Cushion, temperature, in exothermic reactions 2-13
Cylinders, compressed gas
 CGA connections 7-6, 7-8
 discharge example 7-9
 estimating contents 7-3
 heating safely 7-9
 recommended torque values 7-7
 safe handling 7-2
 tips for use 7-2
 types and specifications 7-4
 typical markings 7-4
Cylinders, hazardous reagent 2-11

D

Δ TLM 4-8
 chart for estimating 4-10
DCS (distributed control system) 5-25
De Dietrich, Inc. 2-4
Dead band, control 5-25
Dean-Stark apparatus 2-14
Decible noise scale 9-12
Decimal equivalents, inch fractions 11-11
Decolorization 2-18
Decomposition
 in exothermic reactions 1-6
Dehydration reactions 2-14, 8-20
Delta connection, transformers 5-3
Denatured ethanol 6-44
Density
 comparison of scales 8-17
 of acid-base solutions 8-15
 of inorganic solutions 8-16
 vs. specific gravity 8-17
Depth filters 2-18, 2-26
Derivative control 5-25
Developing scalable reactions 1-7
Dew point vs. relative humidity 7-15
Diastereomers
 calculations for asymmetric crystallization of 11-7
Diatomaceous earth 2-17, 2-18
Dielectric constant 6-5
Differential scanning calorimetry 1-9
Dilutions
 acids and bases 8-4, 8-5
 general 8-5
Dimensional analysis 2-9
Dimensionless groups 2-9

- Disposal, waste streams 9-10
- Distillation 2-19
 and azeotropes 6-25
 atmospheric 2-19
 batch 2-19
 operating tips 2-19
 timeline diagram 2-20
 vacuum 2-19
- Distributed control system (DCS) 5-25
- Documentation, in GMP 1-16
- DOT
 hazards classification 9-8
 labeling requirements 9-8
- Drums
 charging from 2-11
 closure types 3-30
 liquid storage 3-30
 volume of partially filled 3-30
- Dry chemical extinguishers 9-7
- Dry ice 11-11
- Dryers
 classification 2-29
 conductive 2-29
 contact 2-29
 cycle time 2-30
 operating tips 2-32
 product characteristics 2-30
 selection 2-30
 typical setup diagram 2-32
 vacuum 2-30
- Drying
 and scale-up 1-4
 curves, product 2-31
 cycle time 2-30
 energy efficiency of 2-29
 energy requirements 2-32
 major resistances 2-31
 product 2-29
 setting product specifications 2-29
 solvents and solutions 2-18
 spray 2-29
 stages of 2-30
- Drying agents 8-18
- Drying equipment 2-29
- Drying oven setup diagram 2-32
- Drying specification 2-30
- Drying studies 2-30
- Dryness, evaporating to 1-4, 1-6
- DSC 1-9
- Dust explosions 2-12
- Duty cycle controllers 5-13
- Dynamic similarity, in mixing 2-8
- E**
- Elastomers, properties 10-2
- Electric motors 5-11
 classification 5-11
- horsepower 5-11
- nameplate data 5-11
- NEMA frame sizes 5-14
- troubleshooting guide 5-14
- typical current draw 5-12
- voltage 5-11
- Electrical connections, tips 5-5
- Electrical enclosures 5-7
- Electrical safety 5-2
- Electricity
 common voltages 5-3
 single phase 5-3
 three-phase 5-3
 transformers 5-3
 useful formulae 5-3
- Emergency response plan 9-3
- Emulsification
 and work-up 1-8
- Enantiomers
 calculations for asymmetric crystallization of 11-7
- Enclosures
 IP 5-7
 motor 5-12
 NEMA 5-7
- Energy balances 1-14
- Energy dissipation, in mixing 2-10
- Energy dissipation, mixing 2-9
 scale-down example 2-10
- Energy requirements, drying 2-32
- Engineering, in R&D 1-7
- English engineering units 11-4
- Enthalpy of solution 8-14
- Enthalpy of Vaporization
 vs. boiling point 6-18
- Enthalpy of vaporization
 gases 7-11
 solvents 6-5
 vs. temperature 6-19
- Envelope gaskets 3-20
- EPA (Env. Protection Agency) 9-10
- Equations
 for asymmetric crystallization 11-7
 for exponents and logarithms 11-5
 for polynomials 11-5
- Equipment
 cleaning 2-34
 explosion-proof, markings 5-9
 for hazardous locations 5-8
 installation 2-3
 maintenance 2-3
 max. surface temperature codes 5-9
 pilot plant 2-2
 selection 2-3
 used 2-3
- Equipment qualification 1-16
- Equipment train 2-2
- Esterification reactions 8-20
- Ethylene glycol
 boiling point 4-21
 freezing point 4-20
 specific gravity 4-19
 specific heat 4-22
 viscosity chart 4-23
- Eutectic mixtures, cold 11-11
- Evaporating to dryness 1-4, 1-6
- Exothermic reactions
 calorimetry 1-9
 control example 2-13
 decomposition in 1-6
 scale up 2-13
- Expanded pilot time scale 1-3
- Expansion tank 4-11
- Explosion, dust cloud 2-12
- Explosion groups 5-9
- Explosion-proof
 equipment, markings 5-9
 receptacles 5-6
- Explosive limits in air 9-6
- Exponents, equations 11-5
- Exposure limits, chemical 9-6
- Extinguishers, fire 9-7
- Extract phase 2-17
- Extraction
 effectiveness 2-17
 optimization 2-16
- Eye protection 9-12
- F**
- Fabrics, filter cloth 2-26
- Falling rate drying 2-30
- FDA-limited solvents 6-44
- Feed and bleed calculations 11-7
- Ferrules, in fittings 3-15
- Filter aids 2-26
 for carbon removal 2-18
- Filter cake permeability test 2-27
- Filter cloth 2-26
 blinding 2-26
 fabrics 2-26
 permeability 2-26
 porosity 2-26
- Filter-dryers 2-24, 2-25
- Filterite 2-18
- Filters
 Nutsche 2-24
 operating tips 2-24
 pressure 2-24
 Rosenmund 2-24
 rotating drum 2-24
 vacuum 2-24
- Filtrate 2-24
- Filtration
 bench pressure-filter tests 2-27

for product isolation 2-24
 scale-up 2-27
 scale-up example 2-28
 settling test 2-27
 specific cake resistance 2-27
 Strauss equation 2-27
 studies 1-8
 Tiller equation 2-27
 tips for pilot scale 2-28
 Fire extinguishers 9-7
 Fire safety 9-6
 Fittings
 air pressure drop in 7-19
 liquid pressure drop in 3-24
 Flammability
 limits in air, gases 7-11
 solvents, relative 6-3
 Flammable liquids
 charging 2-11
 classification 9-5
 Flanges 3-19
 assembly 3-20
 bolting pattern 3-19
 dimensions 3-19
 pressure rating 3-19
 Flash heating 2-23
 Flash point 6-5, 9-7
 Flat-blade turbine impeller 2-8
 Float switches 5-22
 Flow measurement 5-20
 Flow meters
 Coriolis-type 5-21
 differential pressure 5-20
 magmeters 5-21
 major types 5-20
 positive displacement 5-21
 rotameters 5-20
 selection 5-20
 totalizing 2-11
 turbine 5-21
 vortex 5-21
 Flow number
 in mixing 2-9, 2-10
 vs. Reynold's number 2-9
 Flowrate
 gases through orifice 7-20
 liquids through orifice 3-23
 Fluoropolymers, properties 10-3
 Flush-bottom valve 2-7
 Formylation reactions 8-20
 Friedel-Crafts reactions 8-20
 Functional groups in chemistry 8-19
 Fuses 5-2

G

Galvanic corrosion 10-6
 Galvanic series of metals 10-7

Gas leak detection 7-13
 Gas pressure drop in fittings 7-19
 Gases
 charging to reactors 2-12
 physical properties 7-11
 Gaskets
 envelope 3-20
 flange 3-19
 metal spiral-wound 3-20
 Gauge pressure 5-18
 GC 1-8, 2-29
 gc, proportionality constant 11-4
 Generator status, waste 9-10
 Geometric formulae 11-6
 Geometric similarity, in mixing 2-8
 Glass
 borosilicate 10-8
 corrosion 10-8
 corrosion by acids 10-9
 corrosion by bases 10-9
 corrosion, effect of pH 10-8
 isocorrosion charts 10-8
 limitations of 2-4
 properties of 10-8
 reactors 2-4
 substances known to attack 10-8
 temperature differential limits 2-4
 Glass joints, ground 3-28
 Glass point, glycols 4-18
 Glass-lined steel
 equipment 2-7
 pipe 3-17
 reactors 2-4, 2-5
 Glasses, safety 9-12
 Glove selection guide 9-13
 Glycols 4-18
 boiling point chart 4-21
 concentration formulae 4-18
 ethylene, viscosity chart 4-23
 freezing point chart 4-20
 glass point 4-18
 glass point chart 4-20
 inhibited 4-18
 specific gravity chart 4-19
 specific heat chart 4-22
 water dilution formulae 4-18
 Glyme 6-4
 GMP (Good Manuf. Practice) 1-16
 Government agency acronyms 11-13
 Gravitational proportionality constant, gc 11-4, 11-5
 Ground glass joints 3-28
 Grounding 5-2, 6-2
 Groups, chemical functional 8-19

H

Halocarbon refrigerants 4-27

Halogenated solvents
 environmental impact 1-6
 Halogenation reactions 8-20
 Hand protection 9-13
 Hastelloy 10-5
 reactors 2-5
 substances known to attack 10-28
 Haz-Op 1-10
 and scale-up 1-5
 documenting 1-11
 useful documentation 1-10
 Hazardous location classifications 5-8
 Hazardous materials identification system (HMIS) 9-8
 Hazardous reagents, charging 2-11
 Hazardous substances classification 9-5
 Hazards and Operability Study (Haz-Op) 1-10
 HCFCs 4-27
 Head 3-2, 3-3
 friction 3-3
 pressure 3-3
 pumping 3-2
 static 3-3
 total dynamic 3-2
 velocity 3-3
 Head-capacity curve 3-3
 Hearing protection 9-12
 typical noises levels 9-12
 Heat exchangers 4-8
 example problem 4-9
 temperature profiles 4-8
 Heat of reaction 2-13
 Heat of solution 8-14
 Heat of Vaporization – *See Enthalpy of Vaporization*
 Heat setting, filter cloth 2-27
 Heat transfer
 and scale up 1-3, 1-8
 area vs. volume 4-4
 batch profiles 4-3
 calculations for vessels 4-2
 in stirred reactors 4-2
 isothermal 4-2
 Heat transfer coefficient
 chart for estimating 4-6
 effect of batch properties 4-6
 estimating 4-5
 estimating, example 4-5
 experimental determination 4-7
 measurement, example 4-7
 typical, vessels 4-5
 Heat transfer fluids 4-13
 alcohol, freezing point chart 4-24
 leaking 4-13
 major types 4-14
 organic 4-13
 properties table 4-16

selection 4-14
 specific heat, chart 4-15
 steam 4-25
 Heinkel 2-25
 HEL, Inc. 1-9
 Henderson-Hasselbalch equation 8-8
 High temperature cutoff 4-12
 Hildebrand solubility parameter 6-5, 6-16
 HMIS (Hazardous Materials ID System) 9-8
 Hose
 fittings 3-22
 fittings, sanitary 3-22
 for flammable solvents 3-22
 installation and use 3-22
 Hydration reactions 8-20
 Hydrogen gas
 in hydrogenation reactions 2-14
 special precautions 7-3
 Hydrogenation reactions 8-20
 Hysteresis 5-25

I

IAFIS 3-22
 Ideal gas constant (R) 11-5
 IDLH 9-6
 IEC hazardous zone classification 5-9
 Impeller
 anchor 2-8
 characteristics 2-9
 crowfoot 2-8
 Cryo-Lock 2-8
 curved-blade turbine 2-8
 design 2-8
 flat-blade turbine 2-8
 power number 2-7
 retreat-curve (RCI) 2-8
 types, diagram 2-9
 typical flow numbers 2-9
 typical power numbers 2-9
 Inch fractions to decimal 11-11
 Incompatible chemicals 9-9
 Industrial hose 3-22
 Inert atmosphere 1-7
 Ingress protection (IP) 5-7
 Installation
 equipment 2-3
 pumps 3-5
 Installation qualification (IQ) 2-3
 Insulation, for TCUs 4-11
 Integral control 5-25
 Intermediate specifications 1-16
 Intrinsic safety 5-10
 Intrinsically safe equipment 5-10
 Inventory

control 1-16
 spare parts 2-3
 Ions, names 8-19
 IPS (iron pipe size) 3-14
 IQ (installation qualification) 2-3
 IQ/OQ 2-3, 2-7
 IS (intrinsic safety) 5-10
 ISO pipe standard 3-14
 Isolation barrier 5-10
 Isolation, product 2-24
 Isopropyl ether 6-4
 Isothermal calorimetry 1-9
 Isothermal cooling profile 4-4
 Isothermal heating 4-2

J

Jacket
 heating and cooling 4-2
 temperature during controlled additions 2-13
 Joints, ground glass 3-28

K

Katema, Inc. 2-27
 Kilo-lab 2-2
 Kinematic similarity, in mixing 2-8
 Klinecicz equation 6-19

L

Labeling requirements, chemicals 9-8
 Lantern 2-2, 2-16
 Laser particle detectors 2-21
 Latent heat 4-26
 LD50 9-6
 Leak detection, gases 7-13
 LEL (lower explosion limit) 1-9
 Level calibration, reactor 2-7
 Level measurement 5-22
 Level sensors 5-22
 Library, safety 9-3
 Lift, static suction 3-3
 Lightnin A-310 impeller 2-9
 Limiting oxygen concentration (LOC) 1-9
 Limiting reagent controlled addition 2-13
 Limits, operating volume 1-8
 Limpet coils 4-2
 Liquefied gases 7-11
 Liquid helium 7-3
 Liquid nitrogen 11-11
 Liquid oxygen 7-3
 Liquid ring pumps 7-21

Liquid storage tank capacity 3-29
 Liquid totes 3-30
 Liquid velocity in pipes 3-23
 Load cells 5-22
 LOC (limiting oxygen concn.) 1-9
 Lockout-tagout 5-2, 9-3
 LOD test (loss-on-drying) 2-29
 Log book
 equipment 1-5
 sample 1-5
 Log mean temperature difference 4-8
 chart for estimating 4-10
 Log sheet, batch 1-14
 Logarithms, equations 11-5
 Loss-on-drying test (LOD) 2-29
 Lower explosion limit (LEL) 1-9

M

Magmeters 5-21
 Maintenance
 electrical equipment 5-2
 general equipment 2-3
 pumps 3-6
 reactor 2-7
 Marine impeller 2-9
 Martin Glass Co. 2-4
 Martin reactor, diagram 2-6
 Mass balance 1-14
 Mass transfer, in catalytic hydrogenation 2-14
 Master production record (MPR) 1-14
 Material control 1-16
 Material safety data sheets 9-4
 Materials compatibility table 10-10
 Mathematical constants 11-5
 Mathematical relationships 11-5
 Maximum tolerated dose (MTD) 9-6
 McLeod gauges 5-19
 MeCl_2 [CH_2Cl_2] in extractions 2-17
 Met-L-X extinguishers 9-7
 Metal
 reactor vessels 2-4
 tubing, sizing guide 3-16
 Metals
 galvanic series 10-7
 passivation 10-7
 properties table 10-5
 Metering pumps 2-11
 Methylene chloride,
 environmental impact of 1-6
 Metric tubing, sizing guide 3-16
 Metric units 11-4
 MgSO_4 2-18
 MIE (minimum ignition energy) 1-9
 Millivolts vs. pH 5-24

Minimum ignition energy 1-9
 Minimum ignition temperature 1-9
 Mixer power chart 2-8
 Mixer speed calibration 2-7
 Mixing
 and heterogeneous mixtures 1-4
 and scale up 1-3, 1-8
 energy dissipation 2-10
 in crystallization 2-23
 in distillation 2-20
 power number 2-9
 scale-down 2-8
 scale-down example 2-10
 scale-up 2-8
 tip speed 2-10
 Molar solutions
 acids and bases 8-4
 conversion to weight % 8-15
 vs. normality 8-17
 Monitoring reactions 1-8
 Mother liquors 2-24
 Motors, air 7-17
 Motors, electric 5-11
 codes 5-12
 control 5-13
 efficiency 5-12
 enclosure types 5-12
 frame sizes 5-13
 horsepower 5-11
 kVA values 5-12
 nameplate data 5-11
 NEMA enclosures 5-13
 speed 5-13
 speed controls 5-14
 starters 5-13
 temperature and insulation class 5-13
 troubleshooting guide 5-14
 typical current draw 5-12
 voltage 5-11
 Moyno pump 3-11
 MPR (master production record) 1-14
 MSDS (material safety data sheet) 9-4
 MTD (maximum tolerated dose) 9-6

N

Napping, filter cloth 2-27
 NEMA
 electrical enclosures 5-7
 motor enclosures 5-12
 motor frames 5-13
 Net positive suction head (NPSH) 3-3
 NFPA
 hazard identification system 9-8
 solvent flammability classification 6-2
 Nitration reactions 8-20
 Noise levels 9-12
 Nomenclature

common reaction types 8-20
 common substituents 8-19
 ion names 8-19
 organic functional groups 8-19
 Normal solutions, acids and bases 8-4
 NPSH (net positive suction head) 3-3
 NPT (national pipe thread) 3-14
 Nucleation, in crystallization 2-23
 Nutche filters 2-24

O

O-ring size chart 3-27
 OD tubing
 fittings 3-15
 metal 3-16
 metric 3-16
 OHC (overall heat transfer coeff.) 2-7
 Ohm's law 5-4
 Oil, vacuum pump 7-22
 Open cup testing, flashpoint 1-9
 Operating limits 1-7
 Operating philosophy, pilot plant 1-5
 Operating volume
 in PFD 1-13
 limits 1-8
 OQ (operational qualification) 2-3
 Organic chemistry
 common substituents 8-19
 functional groups 8-19
 reaction types 8-20
 Organization acronyms 11-13
 Organoboranes 2-11
 Organolithium reagents 2-11
 Orifices
 air flow rate 7-20
 liquid flow rate 3-23
 Oven, drying, setup diagram 2-32
 Overall heat transfer coefficient 2-7, 4-5
 Oxidation reactions 8-20
 Oxidizers, classification 9-5

P

P&ID (piping and instrumentation diagram) 2-2
 Paddle impeller 2-9
 Pall filters 2-18
 Parker fittings 3-15
 Particulates filter
 for vacuum pumps 2-12
 Partition coefficient 2-16
 Passivation, metal 10-7
 PEL (permissible exposure limit) 9-6
 Periodic table of the elements 8-2
 Peristaltic pumps 3-11
 tubing relative life, chart 3-12
 tubing selection 3-11
 tubing size and flowrate 3-12
 tubing sizing guide 3-13
 Perlite 2-18
 Permeability
 filter cake 2-27
 filter cloth 2-26
 Permissible exposure limit (PEL) 9-6
 Peroxides
 explosive 6-2
 organic, classification 9-5
 Personal protective equipment
 eye and face 9-12
 glove selection guide 9-13
 hearing protection 9-12
 respirators 9-16
 Personnel training 1-16
 Pfaudler, Inc. 2-4, 2-8
 reactor diagram 2-5
 PFD (process flow diagram) 1-13
 pH
 acid, base solutions 8-3
 color indicators 8-9
 control 5-23
 control problems 5-24
 measurement 5-23
 meter calibration 5-24
 pH scale 5-23
 probes 5-23
 probes, care of 5-24
 reference electrode 5-23
 temperature compensation 5-23
 vs. millivolts 5-24
 Phase separations 2-16, 2-17
 reverse 1-6
 Phosphate buffer 8-8
 Physical constants 11-5
 PID controllers 5-25, 5-26
 Pilot plant, functions of 1-2
 Pipe
 air flow in 7-19
 borosilicate glass 3-17
 BPT 3-14
 copper 3-16
 CPVC 3-18
 dope 3-15
 fittings, pressure drop 3-24
 glass-lined steel 3-17
 ISO 3-14
 JPT 3-14
 liquid velocity in 3-23
 NPT 3-14
 NPT, sizing charts 3-14
 plastic 3-18
 plastic, pressure drop 3-18
 pressure drop 3-24
 pressure drop, viscosity correction 3-25
 PVC 3-18

- schedule 3-14
 - soldering 3-16
 - threaded 3-14
 - threaded, assembly 3-15
 - Piping and instrumentation diagram (P&ID) 2-2
 - Pitched-blade turbine impeller 2-8
 - pKa
 - of acids in water 8-6
 - of amino acids in water 8-7
 - of bases in water 8-7
 - of buffers in water 8-7
 - Plastics
 - pipe 3-18
 - properties 10-4
 - recycling symbols 10-4
 - PLC (programmable logic controller) 5-25
 - Poisons, classification 9-5
 - Polarized outlet 5-6
 - Polish filtration 2-17, 2-18
 - Polymers, properties 10-2, 10-4
 - Polymorphism 2-21
 - Porosity, filter cloth 2-26
 - Powder and dust testing 1-9
 - Powders
 - charging to reactors 2-12
 - Powders and dusts 1-9
 - Power draw, mixers 2-2
 - Power number, mixing 2-7, 2-9
 - vs. Reynold's number 2-9
 - ppm vs. weight % 8-17
 - Precipitation 2-22
 - Pressure conversion diagram 11-9
 - Pressure drop
 - air, in fittings 7-19
 - liquids, in fittings 3-24
 - liquids, in pipe 3-24
 - Pressure filters 2-24
 - tests 2-27
 - Pressure gauges
 - accuracy standards 5-18
 - bellows-type 5-18
 - Bourdon tube type 5-18
 - dial 5-18
 - liquid filled 5-18
 - precautions 5-18
 - protective devices 5-19
 - valve manifold for 5-19
 - Pressure head 3-3
 - Pressure measurement 5-18
 - Pressure rating
 - ASME 2-6
 - vessels 2-6
 - Pressure regulators
 - gas 7-10
 - liquid 3-21
 - Pressure relief
 - rupture disk 2-7
 - valves 3-21
 - vents, calorimetry and sizing 1-9
 - Pressure sensors
 - Bourdon tubes 5-18
 - diaphragm 5-19
 - electronic 5-19
 - gauges 5-18
 - protective devices 5-19
 - Process chillers 4-26
 - Process control 5-25
 - Process efficiency and scale up 1-7
 - Process flow diagram (PFD) 1-13
 - Process safety information (PSI) 1-10
 - Process safety screening 1-9
 - Process validation, in GMP 1-16
 - Product centrifuges 2-25
 - Product drying
 - cycle time 2-30
 - drying curves 2-31
 - material characteristics 2-30
 - Product isolation 2-24
 - Product liquors 2-24
 - Product specifications 1-8, 1-16
 - Programmable logic controller 5-25
 - Proportional control 5-25
 - psia 5-18
 - psig 5-18
 - PTFE 10-3
 - lined vessels 2-5
 - Pumping number, in mixing 2-10
 - Pumps
 - best efficiency point 3-3
 - cavitation 3-3
 - centrifugal 3-7
 - affinity laws 3-4
 - impeller design 3-7
 - performance curves 3-7
 - viscosity correction chart 3-26
 - classification 3-2
 - continuous cavity 3-11
 - diaphragm 3-8
 - installation and operating tips 3-8
 - troubleshooting guide 3-8
 - viscosity correction chart 3-26
 - dynamic 3-2
 - efficiency 3-3
 - external vane 3-10
 - flexible impeller 3-9
 - flexible liner 3-10
 - gear 3-9
 - head capacity curve, figure 3-3
 - head, definition 3-2
 - head, diagram 3-3
 - head/capacity curve 3-3
 - installation 3-5
 - liquid ring 7-21
 - maintenance 3-6
 - metering 2-11
 - diaphragm 3-8
 - piston 3-9
 - motor size 3-4
 - Moyno 3-11
 - operating costs 3-4
 - peristaltic 3-11
 - tubing life, chart 3-12
 - tubing selection 3-11
 - tubing size and flowrate 3-12
 - tubing sizing guide 3-13
 - piston metering 3-9
 - positive displacement 3-2
 - power requirements, chart 3-4
 - progressive cavity 3-11
 - rotary lobe 3-10
 - rotary, troubleshooting guide 3-6
 - rotary vane 3-10
 - safe operation 3-5
 - selection 3-4
 - sizing 3-4
 - system curve 3-3
 - terminology 3-2
 - throttling discharge 3-5
 - typical installation, diagram 3-5
 - vacuum 7-21
- Purity, chemical grades 8-18
- Pyrophoric reagents, charging 2-11

Q

- Qualification
 - equipment, in GMP 1-16
 - installation 2-3
 - operational 2-3
- Quality attributes 1-8
- Quality control 1-16
- Quenching agents 2-16
- Quenching reactions 2-16

R

- R&D in scale-up 1-16
- Radar level measurement 5-22
- Raffinate phase 2-17
- Ramping temperature 4-3
- Raw materials
 - calculating 1-14
 - charge ranges 1-4, 1-7
 - charging 2-11
 - charging, in scale up 1-4
 - controlled addition 2-11
 - hazardous, charging 2-11
 - hazards of 1-7
 - list 1-14
 - specifications 1-16
 - use test 1-5
- RCI (retreat curve impeller) 2-8

Index

- RCRA (Resource Conservation and Recovery Act) 9-10
- Re-glassing vessels 2-7
- React-IR 2-14
- Reaction hazards and scale-up 1-8
- Reactions
- calorimetry 1-9
 - common types 8-20
 - control 1-4, 2-13
 - dehydration 2-14
 - end point determination 2-13
 - exothermic
 - calorimetry 1-9
 - decomposition in 1-6
 - scale up 2-13
 - kinetics 2-13
 - monitoring 1-8
 - quenching 2-16
 - rate control 2-13
 - telescoping 1-7
- Reactive reagents, charging 2-11
- Reactor train 2-2
- Reactors 2-4
- access 1-4
 - agitator selection 2-7
 - automated bench 2-21
 - batch 2-4
 - Buchi 2-4, 2-6
 - cleaning 2-34
 - continuous 2-4
 - falling film 2-4
 - glass 2-4
 - glass-lined 2-4, 2-7
 - diagram 2-5
 - patching 2-7
 - re-glassing 2-7
 - Hastelloy 2-5
 - heat transfer 4-2
 - heat transfer coefficient 4-5
 - height-to-diameter ratio 2-4, 2-8
 - installation 2-7
 - jacketed 4-2
 - level calibration 2-7
 - maintenance 2-7
 - Martin glass 2-6
 - metal 2-4
 - mixing
 - and scale up 1-3
 - baffles 2-4
 - power chart 2-8
 - speed calibration 2-7
 - plug-flow 2-4
 - sampling 2-15
 - size determination 2-5
 - typical services 2-7
 - visibility in 1-4
 - volume calibration 2-7
- Reagent accumulation 2-13
- Reagent addition 1-7
- Reagents, hazardous, charging 2-11
- Receptacles, electrical 5-2
- common configurations 5-6
 - hazardous location 5-6
 - polarized 5-6
- Reduced pressure boiling point 6-22
- Reference electrode, pH 5-23
- Reference pressure 5-18
- Reflux
- for heat removal 2-14
 - operating at full 1-8
- Refractive index 1-8, 6-5
- Refrigerants 4-27
- Refrigeration 4-27
- Regulators, pressure
- gas 7-10
 - liquid 3-21
- Relative humidity vs. dew point 7-15
- Report, campaign 1-15
- Resistor color code 5-5
- Reslurry in crystallization 1-8
- Resource Conservation and Recovery Act (RCRA) 9-10
- Respirators 9-16
- Retain samples 1-5
- Reverse phase splits 1-6
- Rework, in crystallization 1-8
- Reynold's number, mixing 2-9
- Rosenmund 2-24, 2-25
- Rotameters 5-20
- Rotating drum filters 2-24
- RTD temperature probes 5-16
- Rubber, properties 10-2
- Rubber stoppers
- dimensions 3-28
 - sizing chart 3-28
- Rupture disk 2-7
- S**
- SAE fittings 3-15
- Safety
- chemical hygiene plan 9-3
 - committee 9-3
 - fire 9-6
 - glasses 9-12
 - glove selection guide 9-13
 - hand protection 9-13
 - handling chemicals 9-2
 - health and toxicity 9-6
 - hearing protection 9-12
 - library 9-3
 - respiratory protection 9-16
- Safety screening, process 1-9
- Sample log book 1-5
- Samples, retain 1-5
- Sampling
- apparatus 2-15
 - for reaction completion 2-15
 - via manway 2-15
- Sanitary hose fittings 3-22
- Saturated solutions 8-10
- Saybolt viscosity units 3-25
- Scalable reactions
- tips for developing 1-7
- Scale-up, major factors in 1-3
- SCBA 9-16
- Schedule, pipe 3-14
- Scrubbers 2-33
- Scrubbing solutions 2-33
- SDA-3 ethanol 6-44
- Seebeck effect 5-15
- Seeding crystallizations 2-23
- Sefar 2-27
- Sensible heat 4-26
- Service factor, motors 5-12
- Setpoints, control 5-25
- Settling test for filtration 2-27
- Shipping label requirements 9-8
- Shock sensitivity testing 1-9
- Short term exposure limit (STEL) 9-6
- Shuttles, liquid 3-30
- SI units 11-4
- Sieves, specifications 11-12
- Similarity laws, centrifugal pumps 3-4
- Single phase power 5-3
- Slurry density 2-21
- Soft-start controllers 5-13
- Soldering
- electrical 5-5
 - pipe 3-16
- Solids
- charging to reactors 2-12
 - handling hazards 2-12
- Solkafloc 2-18
- Solubility
- aqueous 8-10
 - general rules, inorganics 8-10
 - inorganic compounds, aqueous 8-11
 - organic compounds, aqueous 8-12
 - tips for measuring 8-10
 - USP definitions of 8-10
 - vs. temperature 8-13
- Solubility curve, in crystallizations 2-21
- Solubility map of common solvents 6-16
- Solution
- enthalpy of 8-14
 - heat of 8-14
- Solutions
- charging 2-11
 - density
 - acids and bases 8-15
 - inorganic compounds 8-16
 - for scrubbers 2-33

- Solvation
in crystallization 2-21
- Solvents
binary azeotrope tables 6-28
boiling point vs. pressure 6-23
boiling point at reduced pressure 6-22
charging 2-11
critical temperature 6-19
drying 2-18
drying agents for 8-18
enthalpy of vaporization vs. temperature 6-19
flashpoints 6-3
for crystallization 6-4
halogenated 1-6
NFPA flammability classification 6-2
problematic for extractions 2-17
properties table 6-5
recovery 2-21
relative solubility map 6-16
safe handling 6-2
selection 6-4
solvent exchange 2-19
specific heat vs. temperature 6-21
ternary water-containing azeotrope tables 6-40
vapor pressure at elevated temperature 6-24
viscosity vs. temperature 6-20
- Spade connectors 5-5
- Spare parts 2-3
- Sparkler, Inc. 2-18
- Specific cake resistance 2-27
- Specific gravity vs. density 8-17
- Specific heat 6-5
of HTFs vs. temperature 4-15
solvents vs. temperature 6-21
- Specific volume vs. temperature 6-17
- Specifications
product 1-8, 1-16
product drying 2-29
quality 1-16
raw materials/intermediates 1-16
- Sponge balls 2-34
- Spontaneous combustion 9-7
- Spray ball 2-34
- Spray drying 2-29
- Spreadsheet, PFD 1-13
- Stability
crystal slurry 2-22
reagent solutions 1-8
- Stainless steel
properties 10-5
reactors 2-4
substances known to attack 10-28
- Steam
pressure-temperature chart 4-25
tracing 1-6
- STEL (short term exposure limit) 9-6
- Stoppers, rubber
dimensions 3-28
sizing chart 3-28
- Storage drums 3-30
volume of partially filled 3-30
- Storage tanks
capacity 3-29
volume of partially-filled 3-29
- Straight thread fittings 3-15
- Strain gauges 5-19
- Sulfonation reactions 8-20
- Superalloys 10-5
- Supersaturation 2-22
- Swagelok fittings 3-15
- System curve, pumps 3-3
- Système International units 11-4
- T**
- Tanks
capacity 3-29
volume of partially-filled 3-29
- Tantalum
patch for reactors 2-7
properties 10-5
substances known to attack 10-28
- TCUs 4-11
- Teflon 10-3
- Teflon tape 3-15
- Telescope reactions 1-7
- Temperature
compensation, pH 5-23
control and scale up 1-3
control units 4-11
heat transfer fluids for 4-13
operating tips 4-12
schematic 4-11
selection and design 4-12
conversion diagram 11-8
max differential for glass reactors 2-4
measurement 5-15
ramping in vessels 4-3
sensors
in intrinsically safe circuits 5-17
installation and use 5-17
typical configurations 5-16
- Templates, downloadable 1-13
- Terminal connectors 5-5
- TGA 2-29
- Thermal conductivity
glass 10-8
heat transfer fluids 4-16
metals 10-5
- Thermal runaway 1-6
- Thermal shock
glass 2-4
glass-lined steel pipe 3-17
- Therminol 4-15
- Thermistors 5-16
- Thermocouples 5-15, 5-16
- Thermometers
bimetal 5-15
liquid filled 5-15
- Thermostats 5-25
- Things to avoid in scale-up 1-6
- Things to do during scale-up 1-5
- Threaded pipe 3-14
- Three-phase power 5-3
- Threshold limit value (TLV) 9-6
- Tiller equation for filtration 2-27
- Time scale, pilot 1-3, 1-7
- Timeline, distillation 2-20
- Tip speed, impeller 2-9, 2-10
and shear 1-4
- TLV (threshold limit value) 9-6
- To do, list of 12 things 1-5
- Ton, in refrigeration 4-27
- Torque meter, mixer 2-2
- Total dynamic head 3-2
- Totalizing flow meter 2-11
- Totes, liquid 3-30
- Toxic Substances Control Act (TSCA) 9-10
- Toxicity classifications 9-6
- Training, personnel 1-16, 9-3
- Transformer, electrical 5-3
- Transmitter, 4-20 mA 5-17
- Tray dryer setup diagram 2-32
- Tri-clamp fittings 3-22
- Trigonometric formulae 11-7
- Troubleshooting guides
diaphragm pumps 3-8
electric motors 5-14
rotary pumps 3-6
- TSCA (Toxic Substances Control Act) 9-10
- Tubing
copper 3-16
metal, OD 3-16
- Turbidity 2-21
- Turbulent flow 2-9
- Twaddell density scale 8-17
- Twelve things to avoid 1-6
- Tycon Technologies 2-4
- U**
- Unit conversion factors 11-2
- Units
common prefixes 11-4
comparison of systems 11-4
conversion factors 11-2
- Use test, raw materials 1-5
- Used equipment 2-3

Index

USP (United States Pharmacopeia)
-grade water 6-47
solubility definitions 8-10
Utilities, pilot plant 2-2

V

Vacuum conversion diagram 11-10
Vacuum distillation 2-19
Vacuum pumps 7-21
 capacity 7-21
 for drying 2-32
 installation and maintenance 7-22
 setup, schematic 7-22
Vacuum sensors 5-19
Vacuum systems 7-21
Validation, process 1-16
Valves 3-20
 ASME-coded 3-21
 ball 3-20
 butterfly 3-20
 check 3-21
 common types 3-20
 diaphragm 3-21
 flush-bottom 2-7
 gate 3-20
 globe 3-20
 needle 3-21
 packings 3-21
 pressure relief 3-21
 solenoid 3-21
 specialty 3-21
Vapor pressure
 at elevated temperature 6-24
 vs. volume % in air 9-6
Variable frequency drive 3-3, 5-14
Velocity head 3-3
Velocity, liquid in pipes 3-23
Venturi meter 5-21
Vessels
 cooling example 4-2
 heat transfer coefficient 4-5
 heating arrangements 4-2
 pressure rating 2-6
 reactor 2-4
Viscosity 6-5
 centrifugal pump correction chart 3-26
 diaphragm pump correction chart 3-26
 gases 7-11
 kinematic, conversion chart 3-25
 pressure drop correction 3-25
 Saybolt units 3-25
 solvents vs. temperature 6-20
 typical liquids 3-25
 water vs. temperature 6-45
Viscous flow 2-9
Visibility, in reactors 1-4, 2-4
Visual inspection, in cleaning 2-34

VLE (vapor liquid equilibrium) diagram 6-25
Voltage drop in wire 5-4
Voltage variations 5-3
Volume calibration, vessels 2-7
Volumetric seals, pressure gauges 5-19

W

Wand, solvent charging 2-11
Waste disposal 9-10
 and DOT 9-10
 common treatment methods 9-11
 documentation requirements 9-11
 effect of cost on process 1-6
 generator classification 9-10
 regulations 9-10
 segregation and storage 9-10
 waste minimization 9-10
Waste stream report 1-14
Water
 boiling point vs. elevation 6-46
 common purification methods 6-47
 conductivity and resistivity 6-48
 density vs. temperature 6-45
 dielectric const. vs. temperature 6-45
 hardness 6-47
 selected properties 6-45
 USP grade 6-47
 viscosity vs. temperature 6-45
Watt meter 2-2
Weight % solutions, acids and bases 8-5
Weight % vs. ppm 8-17
Western States Machine 2-25
Westfalia 2-25
What if?, in Haz-Op 1-11
Wire cloth specifications 11-12
Wire, electrical 5-4
 color code 5-4
 copper
 ratings 5-4
 sizes 5-4
 max allowable length 5-4
 type codes 5-4
 voltage drop 5-4
Wire nuts 5-5
Work-up 1-4, 2-16

Y

Y-connection, transformers 5-3
Yield calculation 1-14

Z

Z87 eyewear 9-12
Zener barrier 5-10

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Published by
FXM ENGINEERING AND DESIGN
Worcester, Massachusetts

For more information visit www.pprbook.com

ISBN 0-9721769-1-8



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5 8 9 9 5

